ANALYTICAL ABSTRACTS

Dealing with all branches of Analytical Chemistry: Published Monthly with THE ANALYST by the Society for Analytical Chemistry

Editor: NORMAN EVERS, B.Sc., Ph.D., F.R.I.C.
14, BELGRAVE SQUARE, LONDON, S.W.1
Telephone: BELgravia 3258

Published for the Society by
W. HEFFER & SONS, LTD., CAMBRIDGE, ENGLAND

Volume 5 Price 8s.
Subscription Rate, inclusive of Index, 75s. per annum, Post Free
No. 11, Abstracts 3584-3960 November, 1958

FOR REVISED PRICES FROM 1st JANUARY, 1959, SEE OVERLEAF

Binding . . .

W. Heffer & Sons Ltd. announce that they are now able to BIND copies of ANALYTICAL ABSTRACTS, in an approved binding case, at a cost of 12/6 per volume. The 12 Parts for 1957 together with Index and remittance for 12/6 should be sent to

W. HEFFER & SONS, LIMITED
HILLS ROAD, CAMBRIDGE

IMPORTANT NOTICE TO SUBSCRIBERS

(Other than Members of the Society)

For 1959 the Subscription to *Analytical Abstracts*, inclusive of Index, will be £5 per annum; and for *Analytical Abstracts* together with *The Analyst* and Indexes will be £8 8s. per annum.

Analytical Abstracts is available printed on one side of the paper only, suitable for incorporation into reference systems; the cost will be £5 7s. 6d. per annum, exclusive of Index.

The price of single copies of *Analytical Abstracts*, including back numbers, will be 15s. post free.

All further enquiries about subscriptions should be made through the Secretary, the Society for Analytical Chemistry, 14, Belgrave Square, London, S.W.1. Telephone: BELgravia 3258.

Now available for your reference library

Recent
Russian
Research in

ION-EXCHANGE CHROMATOGRAPHY

in complete English translation

PUBLISHED IN the Soviet Union, late in 1957, this collection of twenty stimulating papers represents the outstanding contributions to a session convened by the *Commission on Chromatography, Division of Chemical Sciences, Academy of Sciences, USSR.*

As a result, researchers in this field are now furnished with the most advanced Soviet theories on the *application of ion-exchange chromatography*, as well as interesting original research findings and significant reviews of work done on *various applications of ion-exchange resins* particularly in the fields of *food* and *medicine*.

A glance at the complete list of papers shown below is merely an indication of the immense value of this volume for keeping Western scientists fully aware of current Soviet progress in this important field.
English translation, 184 pages, $35.00.

Complete List of Papers:

1. Laminar Method for Approximate Calculation of Chromatograms.
2. Equations for Ion Exchange Isotherms and their Experimental Verification.
3. Absorption of the Cations of Heavy Metals by Anion Exchange Resins.
4. Principles of Adsorption Technology.
5. Laws of Adsorption from Solutions under Dynamic Conditions during Separation of Alkaloids by Means of Ion Exchange Resins.
6. Ion Exchange of Alkaloids on Synthetic Ion Exchange Resins.
7. Structure of Cation Exchange Resins. Their Suggested Chemical Structure, and Possible Breakdown Products.
8. Basic Problems in the Synthesis and Study of Ion Exchange Resins.
9. Physicochemical Properties of Ion Exchange Resins.
10. Properties of some Cation Exchange Resins and the Possibility of Using Them in the Medical and Food Industries.
11. Properties of Certain Anion Exchange Resins and the Possibility of Using Them in the Medical and Food Industries.
12. Purification of Certain Chemical Compounds by Means of Ion Exchange Sorbents.
13. Possibility of Using Ion Exchange Resins for Obtaining Drinking Water of High Mineral Content.
14. Purification of Lactic Acid by Means of Ion Exchangers.
15. Methods for Isolating Anabasine, Spherophysine, and Methyl-Caffeine from Plant Raw Material and Mother Liquors by Means of Ion Exchangers.
16. Elution Curves of Morphine during Ion Exchange Chromatography on Cation Exchangers.
17. Application of Ion Exchange Adsorbents in Pharmaceutical Analysis.
18. Absorption of Streptomycin by the Hydrogen and Hydrogen-Salt Forms of Carboxylic Cation Exchangers.
19. Use of Ion Exchange Sorbents for Preventing Blood Clotting.
20. Separation of Cadmium and Copper, Cadmium and Lead, and Cadmium and Bismuth by Ion Exchange Chromatography.

C.B. translations by bilingual scientists are reproduced by the multilith process from easy-to-read IBM cold type, and include all diagrammatic, tabular and photographic material integral with the text. Books are staple-bound in sturdy paper covers.

REQUEST INFORMATION RE PAYMENT IN STERLING.

At your bookstore, or order directly from:

CONSULTANTS BUREAU, INC.

227 WEST 17th STREET, NEW YORK 11, NEW YORK

Telephone: ALgonquin 5-0713 · Cable Address: CONBUREAU, NEW YORK

Binding . . .

W. Heffer & Sons Ltd. announce that they are now able to BIND copies of ANALYTICAL ABSTRACTS, in an approved binding case, at a cost of 12/6 per volume. The 12 Parts for 1957 together with Index and remittance for 12/6 should be sent to

W. HEFFER & SONS, LIMITED
HILLS ROAD, CAMBRIDGE

IMPORTANT NOTICE TO SUBSCRIBERS

(Other than Members of the Society)

For 1959 the Subscription to *Analytical Abstracts*, inclusive of Index, will be £5 per annum; and for *Analytical Abstracts* together with *The Analyst* and Indexes will be £8 8s. per annum.

Analytical Abstracts is available printed on one side of the paper only, suitable for incorporation into reference systems; the cost will be £5 7s. 6d. per annum, exclusive of Index.

The price of single copies of *Analytical Abstracts*, including back numbers, will be 15s. post free.

All further enquiries about subscriptions should be made through the Secretary, the Society for Analytical Chemistry, 14, Belgrave Square, London, S.W.1. Telephone: BELgravia 3258.

Now available for your reference library—

Recent
Russian
Research in

ION-EXCHANGE CHROMATOGRAPHY

in complete English translation

PUBLISHED IN the Soviet Union, late in 1957, this collection of twenty stimulating papers represents the outstanding contributions to a session convened by the *Commission on Chromatography, Division of Chemical Sciences, Academy of Sciences, USSR.*

As a result, researchers in this field are now furnished with the most advanced Soviet theories on the *application of ion-exchange chromatography*, as well as interesting original research findings and significant reviews of work done on *various applications of ion-exchange resins*—particularly in the fields of *food* and *medicine*.

A glance at the complete list of papers shown below is merely an indication of the immense value of this volume for keeping Western scientists fully aware of current Soviet progress in this important field.
English translation, 184 pages, $35.00.

Complete List of Papers:

1. Laminar Method for Approximate Calculation of Chromatograms.
2. Equations for Ion Exchange Isotherms and their Experimental Verification.
3. Absorption of the Cations of Heavy Metals by Anion Exchange Resins.
4. Principles of Adsorption Technology.
5. Laws of Adsorption from Solutions under Dynamic Conditions during Separation of Alkaloids by Means of Ion Exchange Resins.
6. Ion Exchange of Alkaloids on Synthetic Ion Exchange Resins.
7. Structure of Cation Exchange Resins, Their Suggested Chemical Structure, and Possible Breakdown Products.
8. Basic Problems in the Synthesis and Study of Ion Exchange Resins.
9. Physicochemical Properties of Ion Exchange Resins.
10. Properties of some Cation Exchange Resins and the Possibility of Using Them in the Medical and Food Industries.
11. Properties of Certain Anion Exchange Resins and the Possibility of Using Them in the Medical and Food Industries.
12. Purification of Certain Chemical Compounds by Means of Ion Exchange Sorbents.
13. Possibility of Using Ion Exchange Resins for Obtaining Drinking Water of High Mineral Content.
14. Purification of Lactic Acid by Means of Ion Exchangers.
15. Methods for Isolating Anabasine, Spherophysine, and Methyl Caffeine from Plant Raw Material and Mother Liquors by Means of Ion Exchangers.
16. Elution Curves of Morphine during Ion Exchange Chromatography on Cation Exchangers.
17. Application of Ion Exchange Adsorbents in Pharmaceutical Analysis.
18. Absorption of Streptomycin by the Hydrogen and Hydrogen Salt Forms of Carboxylic Cation Exchangers.
19. Use of Ion Exchange Sorbents for Preventing Blood Clotting.
20. Separation of Cadmium and Copper, Cadmium and Lead, and Cadmium and Bismuth by Ion Exchange Chromatography.

C.B. translations by bilingual scientists are reproduced by the multilith process from easy-to-read IBM cold type, and include all diagrammatic, tabular and photographic material integral with the text. Books are staple-bound in sturdy paper covers.

REQUEST INFORMATION RE PAYMENT IN STERLING.

At your bookstore, or order directly from:

CONSULTANTS BUREAU, INC.

227 WEST 17th STREET, NEW YORK 11, NEW YORK

Telephone: ALgonquin 5-0713 · Cable Address: CONBUREAU, NEW YORK

ANALYTICAL ABSTRACTS

1.—GENERAL ANALYTICAL CHEMISTRY

3584. **Statistics in chemical analysis. II. Evaluation of practical analyses.** G. Gottschalk and P. Dehmel (Osram G.m.b.H., Helmholtzstr. 4–8, Berlin). *Z. anal. Chem.*, 1958, **160** (3), 161–169.—The application of the values obtained for standard deviation, variance and systematic error from model analyses as described in Part I (*Z. anal. Chem.*, 1957, **159**, 81) to practical analyses is set out and explained with examples. Confidence limits for individual and mean results from a single set of determinations made under conditions identical with those of the model analyses can be calculated from the standard deviation after the correction for systematic error has been applied. The requirement of identical conditions must be strictly observed, and with unknowns a preliminary analysis for foreign ions is necessary. Homogeneity of replicates can be tested by comparison of the variance with that of the model analyses. By means of the standard deviation, the means of two sets of determinations on different samples can be compared to determine if a significant difference exists, and sensitivity limits for a procedure can be estimated. The minimum number of replicates to satisfy prescribed confidence limits can be estimated.
G. BURGER

3585. **Improved chemical reagent for the quantitative determination of water.** E. Blomgren and H. Jenner (Aktiebolaget Pharmacia). U. S. Pat. 2,780,601 (5.2.1957).—A stabilised Karl Fischer reagent is described wherein the I^-:I ratio is about 3·2. Pyridinium iodide is the source of I^-, methanol is the solvent and 1·2 mole per litre of SO_2 and 3·5 moles per litre of pyridine are present. A set of curves shows the stability of this reagent over 3 months as compared with an ordinary reagent. CHEM. ABSTR.

3586. **Sodium tetraphenylboron, 1957: a bibliography.** A. J. Barnard, jun., and H. Büechl (J. T. Baker Chem. Co., Phillipsburg, N.J.). *Chemist Analyst*, 1958, **47** (2), 46–47.—This bibliography contains 16 additional references to publications of 1955–1956 and 57 references for 1957. N. E.

3587. **Bromothallic acid as a new reagent for the identification of organic bases.** G. Sandri (Ist. di Chim. Agrar., Univ. Bologna, Italy). *Mikrochim. Acta*, 1958, (2), 253–258.—Descriptions are given of several ppt. given by bromothallic acid with organic bases such as betaine, choline and nicotinamide. Of special interest is the possibility of differentiating between purine bases such as caffeine, theobromine and theophylline. D. F. PHILLIPS

3588. **New colour reactions of phthalein complexone.** R. Belcher, M. A. Leonard and T. S. West (Chem. Dept., The Univ., Birmingham, England). *Chem. & Ind.*, 1958, (5), 128–129.—Colour reactions of phthalein complexone with Mn, Cd, Hg, Ag, Pb, V, Co, Ga and Th are described, with its use, when screened with Xylene Cyanol FF and dimethyl yellow, as an indicator for the titration of Cd and Mn with EDTA. For the determination of Cd, 25 ml of soln. is treated with 4 drops of indicator soln. (suspend 0·5 g in water, dissolve with a little aq. NH_3 and make up to 100 ml), 2 drops of 0·1% Xylene Cyanol FF, 5 drops of 0·1% dimethyl yellow, a few ml of NH_4Cl - NH_3 buffer (pH $\simeq$ 10) and 5 ml of ethanol, and titrated with 0·02 *M* EDTA (disodium salt) to a colour change from a deep red - mauve, through grey, to green. For the determination of Mn the procedure is similar, except that excess of EDTA is added before the buffer, and back-titrated with standard manganese soln., the end-point being the first degradation of the green colour of the screened indicator.
R. E. ESSERY

3589. **Studies on stilbene. XIV. Analytical applications of 4-stilbenylnitrosohydroxylamine, "styrylcupferron."** G. Drefahl and A. Geissler (Inst. für Org. Chem. und Biochem., Friedrich Schiller Univ., Jena). *Z. anal. Chem.*, 1958, **160** (1), 34–38.—The prep. of the ethanolamine salt of 4-stilbenylnitrosohydroxylamine is described. Compared with cupferron, it is more stable, its metal complexes are less soluble, have a stoicheiometric composition and can be dried at 110°. Bivalent Cu is pptd. from hot ammoniacal soln., from soln. containing citric acid or tartaric acid, and from its tetramine complex. Tervalent Fe is best pptd. from neutral soln. at 50°. Excess of reagent can be dissolved by the addition of *iso*propyl alcohol (15% of the total vol.). Aluminium can be pptd. from ammoniacal soln. containing tartrate or citrate or from weakly acid soln. A little hydroxylamine hydrochloride is added to the aluminium soln. on the water bath and the hot reagent is added. The ppt. is stirred with ethanol - pyridine (4:1) before filtering. Alternatively, Al is pptd. from ammoniacal soln. containing tartrate. Ammonium chloride is added before filtering. G. BURGER

3590. **Methyl*cyclo*hexanone, a new solvent for the extraction of uranium and the separation of uranium from thorium.** K. Seidl and M. Beránek (Inst. Technol. Metals, High-School of Chem. Technol., Prague). *Chem. Listy*, 1958, **52** (2), 337–339.—Uranium can be quant. extracted in 15 min. from a mixture of nitrates of U and Th (100 to 500 mg), dissolved in 20 ml of aq. $NaNO_3$ soln., with the use of 20 ml of 2-methyl*cyclo*hexanone. The presence of HNO_3 causes errors. Uranium can be re-extracted from the org. phase with a satd. soln. of Na_2CO_3. The advantages of 2-methyl*cyclo*hexanone as extracting solvent for this separation are discussed. J. ZÝKA

3591. **Qualitative inorganic analysis. V. The interference of borate ion.** O. B. Hayes and J. Winterburn (Handsworth Tech. Coll., Birmingham, England). *Mikrochim. Acta*, 1958, (2), 197–200 (in English).—Serious interference from borate ion arises from a cause which has hitherto been overlooked. Contrary to statements often made, alkaline-earth borates are not pptd. in group 3 because they are soluble in the NH_4Cl present. But group-4 metals (Zn, Co and Mn), which form insoluble borates, are co-pptd. extensively in group 3 when borate is present. Borate must therefore be removed by double evaporation with methanol and HCl before pptg. group-3 metals.

D. F. PHILLIPS

3592. **Qualitative inorganic analysis. VI. Charcoal-block reactions.** R. Belcher, R. Harrison and W. I. Stephen (Univ., Birmingham, England). *Mikrochim. Acta*, 1958, (2), 201–203 (in English).—Suitable reducing flames allow reactions on the block to go to completion without mouth blowing. Such flames are obtained by either of two procedures. (*a*) Draw out a piece of 8-mm glass tubing to a capillary at one end. Bend the capillary to give a right angle. Attach a gas supply to the tube and allow the small blue flame to impinge directly on the test substance contained in a suitable cavity of the block. (*b*) Connect a conventional mouth-blowpipe (jet-hole diameter ≯ 26 s.w.g. wire) to the gas supply at the mouth end. Use the fine jet as in (*a*). *Procedure*—Mix the test substance with 3 to 4 times its bulk of Na_2CO_3 and transfer to a cavity in the block. Rest the block on the bench and allow the reducing flame obtained as in (*a*) or (*b*) to impinge on the mixture until the reaction is judged to be complete (10 to 20 sec.). D. F. PHILLIPS

3593. **Simple metallochromic indicators of the Eriochrome black T type.** B. Buděšínský (Res. Inst. Pharm. and Biochem., Prague). *Chem. Listy*, 1958, **52** (2), 250–254.—The metallochromic properties of 4-*o*-hydroxyphenylazoresorcinol (**I**), 4-(2-hydroxy-5-sulphophenylazo)resorcinol (**II**), 4-(2-hydroxy-4-nitrophenylazo)resorcinol (**III**), 1-*o*-hydroxyphenylazo-2-naphthol (**IV**), 1-(2-hydroxy-4-nitrophenylazo)-2-naphthol (**V**), 1-(2-hydroxy-5-sulphophenylazo)-2-naphthol (**VI**), 2-*o*-hydroxyphenylazo-1:5-dihydroxynaphthalene (**VII**) and 2-(2-hydroxy-5-sulphophenylazo)-1:5-dihydroxynaphthalene (**VIII**) were studied. **IV** and **V** are suitable indicators for Ca and Sr, **V** for Zn and Cd, and **VII** and **VIII** for Mg, Zn, Cd, Pb, Mn and Ca. *Procedure for Ca and Sr*—To a neutral soln. containing 5×10^{-2} to 1 millimole of Ca^{2+} or Sr^{2+} in 50 to 100 ml add *M* NaOH (10 ml), a few mg of **IV** or **V** and titrate the red - orange soln. with 0·05 or 0·1 *M* EDTA (disodium salt) (**IX**) till a red - violet colour appears. *Procedure for Zn or Cd*—Blue - green soln. (50 to 100 ml) containing aq. NH_3 (25%) (5 ml) can be titrated with 0·05 to 0·1 *M* **IX**. The indicator change is from blue - green to red - violet. When titrating Mg, Zn, Cd or Mn with the use of **VII** and **VIII** as indicators and 0·05 to 0·1 *M* **IX** as volumetric reagent, Schwarzenbach's buffer soln. (NH_4Cl - aq. NH_3) (pH 10) or conc. aq. NH_3 must be added for Ca. The colour change is from red - violet to blue. J. ZÝKA

3594. **pH standard solutions in methanol. Preliminary communication.** C. L. de Ligny and P. F. M. Luykx (Univ., Utrecht). *Rec. Trav. Chim. Pays-Bas*, 1958, **77** (2), 154–156 (in English).—Measurements were made of pH values of certain standard buffer soln. in anhyd. and aq. methanol. The following figures were obtained in 100% methanol (*a*) with silver - silver chloride electrodes in alkali chloride soln., and (*b*) with silver - silver bromide electrodes in alkali bromide soln.—0·01 *M* succinic acid - 0·01 *M* Li H succinate, 8·67, 8·63; 0·01 *M* salicylic acid - 0·01 *M* Na salicylate, 7·48, 7·44; and 0·01 *M* oxalic acid - 0·01 *M* ammonium hydrogen oxalate, 5·73, 5·70. M. DAVIS

3595. **Complexometric titrations (chelatometry). XXXVII. Glycinenaphthol violet, a new chelatometric indicator.** B. Buděšínský (Res. Inst. Pharm. and Biochem., Prague). *Chem. Listy*, 1958, **52** (2), 247–249.—Glycinenaphthol violet (4-*p*-nitrophenylazo-2-glycinomethyl-1-naphthol) (**I**) forms red - orange (pH 2 to 6), red - violet (pH 7 to 10) or blue (pH 10·5 to 12) soln. In the presence of Cd^{2+}, Pb^{2+}, Co^{2+}, Ni^{2+}, Cu^{2+} or Mn^{2+} (pH 10·5), the corresponding complex gives a red - violet colour, or, in the presence of Ca^{2+}, blue. **I** does not form complexes with Hg, Be, Sr, Ba, Sc, Al, La, Zr, Th, Sn, Bi, Fe, Pd or Ag. *Procedure for titration of Ni, Co, Cu, Cd and Mn*—To the neutral soln. of the metal (0·05 to 1 millimole) add aq. NH_3 (25%) (20 ml) and a few mg of **I** and titrate with 0·05 *M* EDTA (disodium salt) till the red - violet colour changes to blue. When determining Mn, hydroxylamine hydrochloride must be added to prevent oxidation. Complexing anions (*e.g.*, chlorides in the titration of Cu^{2+}) interfere. J. ZÝKA

3596. **Modern methods of gas analysis.** G. Kainz (2nd Chem. Inst., Univ., Vienna). *Öst. ChemZtg.*, 1958, **59** (3–4), 45–51.—The most important of the newer physical methods of gas analysis are reviewed, and their advantages and disadvantages discussed. The methods include those based on magnetic susceptibility, heat conductivity, mass spectrography, u.v. absorption and i.r. absorption measurements. Finally, adsorption and partition methods of gas chromatography are described as physical means of separation before analysis.

J. H. WATON

3597. **Gas chromatography.** E. Cremer and L. Roselius (Phys.-Chem. Inst., Univ., Innsbruck, Austria). *Angew. Chem.*, 1958, **70** (2), 42–49.—A review is given of the development of gas chromatography, in practice as well as in theory. The theoretical treatment is considered both from the kinetic and statistical thermodynamic standpoints. Attention is drawn to the application of gas chromatography to the determination of the heats of adsorption of gases, and of the water content of an adsorbent. J. H. WATON

3598. **Chromatographic gas analysis.** W. Schuhknecht (Saarbergwerke, St. Ingbert, Saar). *Arch. Eisenhüttenw.*, 1958, **29** (2), 101–106.—A review. E. G. CUMMINS

3599. **Thermodynamic analysis of gases.** V. Ya. Basevich. *Zhur. Fiz. Khim.*, 1957, **31** (5), 1154–1156; *Ref. Zhur., Khim.*, 1958, Abstr. No. 827.—The gas to be analysed passes through a calibrated orifice for a known time into a previously evacuated vessel. The quantity of gas is accurately measured by the increase in pressure in the vessel, and under definite conditions depends only on the composition of the gas, in accordance with the equation $P_e = P(R, k)$, where P_e is the pressure of the gas in the vessel, R is the gas constant, and k is the ratio of the specific heats at const. pressure and vol. Since the equation is only approximate, it

cannot be satisfied by calculated values of P_e, but the instrument should first be calibrated to determine the dependence of P_e on the composition of the gas. The method may be used for the analysis of gas mixtures for one component which is noticeably different from the remainder of the gas as regards specific heat and gas constant.

C. D. KOPKIN

2.—INORGANIC ANALYSIS

3600. **Precipitation of metals with mono- and di-methylarsinic [methylarsonic and dimethylarsinic] acids.** R. Pietsch (Univ., Graz., Austria). *Mikrochim. Acta*, 1958, (2), 220–224.—The pH at which these two acids start to form ppt. has been studied for 41 cations, and the colour, texture and filtration properties of the ppt. are discussed and compared. D. F. PHILLIPS

3601. **Spectral-isotopic method for the determination of hydrogen in metals.** A. N. Zaidel (Phys. Inst., Leningrad State Univ., USSR). *Spectrochim. Acta*, 1958, **10** (4), 369–376.—Place the sample (*e.g.*, 10 g) in an "exchanger" tube of known vol. (*e.g.*, 150 ml), evacuate the exchanger and fill with deuterium. Heat until equilibrium is reached as a result of isotopic exchange between the sample and the surrounding gas. Determine the isotopic composition of a portion of the gas by passage through a high-frequency discharge tube of diameter 1 or 2 mm at a rate of 1 or 2 ml per min. and a pressure of 2 to 5 mm (Hg) and photo-electric measurement of the ratio of the intensities of the Balmer lines of hydrogen and deuterium. Perform a blank determination. For all samples studied, the ratio of the concn. of hydrogen and deuterium is the same in the metal as in the surrounding gas, within experimental error. Relatively large quantities of foreign gases do not interfere. The results agree with those obtained by the vacuum fusion method. A. R. ROGERS

3602. **Determination of the absolute activity of solid tritium samples.** J. Rydberg (Res. Inst. of Nat. Defence, Sundbyberg 4, Sweden). *Acta Chem. Scand.*, 1958, **12** (3), 399–407.—The true specific activity (S) of a sample of tritium-labelled material may be determined for a solid sample in a 2π internal gas flow proportional counter (90% of A + 10% of CH_4) and is given by $S = I/m.f_e.f_r.f_g.f_b.f_s$ disintegrations per minute per gram, where I is the measured activity, m the mass of the sample, f_e the efficiency correction factor ($\simeq 1$ for this counter), f_r the resolution factor ($< 1\%$ and negligible below 5000 c.p.s.), f_g the geometry factor (0·5 with a flat planchette in the sensitive vol.), f_b the back-scattering factor from the backing material, and f_s the self-absorption factor in the sample. The methods of calculating and determining f_b and f_s are discussed and backings of aluminium, stainless steel and platinum are used. The experimental work was carried out with tritium-labelled 4-aminosalicylic acid. Sample wt. of 0·09 to 2000 μg with sample areas of 1·3 to 0·2 sq. cm were used. A fraction of the same sample was also analysed for tritium as H gas in a Geiger - Müller counter and agreement with the corrected determination in solid samples is good. E. J. H. BIRCH

3603. **Use of external cathode counters for the determination of tritium.** S. Monfeuga (Fac. des Sci., 11 rue Pierre Curie, Paris). *Mikrochim. Acta*, 1958, (2), 177–180 (in French).—Three types of disturbance have been observed when using Geiger - Müller counters of the Maze type in the analysis of organic materials containing tritium. These arise, respectively, from traces of O, from traces of mercury vapour, and from the modification of the state of the glass surface constituting the shell of the counter. The precautions necessary to overcome these faults are stated. D. F. PHILLIPS

3604. **Studies in qualitative analysis. VII. Detection of the alkali metals and magnesium.** L. S. Bark and W. F. Jones (Chem. Dept., Coll. of Technol., Derby, England). *Mikrochim. Acta*, 1958, (3), 406–410 (in English).—Two procedures are given for the separation and detection of Mg, Na, K and Li in the soln. after removal of the Zn group. (*i*)—Magnesium is pptd. as $MgCO_3.(NH_4)_2CO_3$ in ethanol and is confirmed with Titan yellow. Five drops of conc. HCl - bromine water (1:1) are added to the filtrate, the mixture is heated to expel ammonium salts, the residue is dissolved in one drop of dil. HCl (1:30), and NaCl plus KCl is pptd. from diethyl ether - ethanol (4:1). The filtrate is evaporated to dryness; any residue is LiCl, in which Li can be confirmed by the periodate test. The ppt. of mixed chlorides is dissolved in H_2O, and tests for Na (with Cu uranyl acetate) and K (with sodium cobaltinitrite) are made separately. (*ii*)—To ensure absence of ammonium salts, K is detected in the sodium carbonate extract prepared for the examination of the anions. Magnesium is separated as in the first method, after removal of the Zn group, whilst Na and Li are tested for in the residual soln. divided into two portions (after part removal of ethanol). The second method is rapid and is recommended.

W. J. BAKER

3605. **The polarographic determination of lithium, rubidium and caesium.** G. Schöber and V. Gutmann (Inst. Gen. Chem., Tech. Coll., Vienna). *Mikrochim. Acta*, 1958, (3), 319–320 (in German).—Lithium may be determined polarographically in 80% *iso*propyl alcohol soln. which is 0·1 *M* in tetraethylammonium hydroxide. The relatively high concn. of conducting salt obviates interference by acetone, and commercial *iso*propyl alcohol can therefore be used without further purification. The $E_{\frac{1}{2}}$ occurs at $-2{\cdot}37$ V (20° to 30°) and the accuracy of the results is $\pm$ 1%. Rubidium and Cs may be determined in the same medium, but by using lithium hydroxide as the conducting salt in 80% *iso*propyl alcohol soln. at a concn. ratio of 1:50, difficulties due to maxima and poor limiting currents are avoided. The $E_{\frac{1}{2}}$ of Cs and Rb in such soln. at 20° to 30° are $-2{\cdot}03$ V and $-1{\cdot}97$ V, respectively. Results are accurate within $\pm$ 0·5%. Only the total content of Rb and Cs when present together in soln. can be determined, but the determination of either Rb or Cs in the presence of Li can be carried out in the presence of 0·1 *M* tetraethylammonium hydroxide soln. in 80% *iso*propyl alcohol. S. M. MARSH

3606. **The analysis of sodium metal and sodium - potassium alloy (collected Capenhurst methods).** Chief Chemist (Chem. Services Dept., U.K.A.E.A., Capenhurst, Lancs.). U.K.A.E.A. (Ind. Group) Rep. IGO–AM/CA–110, 1958, 56 pp.—The report contains recommendations for safety precautions and sampling procedure and details of the following methods of analysis—the determination of total C,

and C due to carbonate, O (as Na_2O), Rb and Cs in sodium metal, and O (as Na_2O), K, Rb and Cs in sodium - potassium alloy. N. E.

3607. **Flame-spectrometric determination of sodium and potassium with a buffering addition of barium chloride.** F. Hegemann, H. Kostyra and B. Pfab (Tech. Hochschule, Munich, Germany). *Glastech. Ber.*, 1957, **30**, 14–17.—For the rapid determination of the alkalis, particularly Na and K in one soln., $BaCl_2$ is used as a "buffer," levelling the mutual interactions of the Na^+ and K^+ in the acetylene flame. For an accuracy within 0·5% of the end result, the determination of Na is not affected if the Na value is different by $\pm$ 60% from that of K. Similarly the K determination is not affected if the K value is different by $<$ 100% from that of Na. The exact amounts of the $BaCl_2$ soln. and the standard soln. to be added (best stored in polyethylene bottles) are given. Test determinations agree with direct analytical determinations of Na and K in mixed alkaline oxalate soln., common household glass, a green iron-containing glass and a lead arsenate glass. The decomposition of the glasses by HF - oxalic acid (*cf.* Hegemann *et al.*, *Anal. Abstr.*, 1956, **3**, 650) proved highly reliable. Iron oxalate does not interfere in the determinations. CHEM. ABSTR.

3608. **Polarographic determination of sodium monoxide plus potassium monoxide in various refractories.** E. V. Ermolaeva and L. A. Korobka. *Byull. Nauchno-Tekh. Inform., Vses. Nauch. Inst. Ogneuporov*, 1957, **2**, 89–93; *Ref. Zhur., Khim.*, 1958, Abstr. No. 4280.—The conditions for the polarographic determination have been studied. Because of the considerable change in the height of the polarographic wave for Na_2O plus K_2O with change in the ratio of Na_2O to K_2O (increasing with the Na_2O content) and temp. (at 15° to 25° a 1° change in temp. gives rise to a 2·8% relative error), it is recommended to polarograph a control soln. of chemically pure Na_2SO_4 and K_2SO_4 in which the ratio of Na_2O to K_2O corresponds to that in the soln. being analysed. When using tetraethylammonium iodide as background, the sample is prepared as in the preparation for the rapid gravimetric method for determining total Na plus K in clay and chamotte (Balyuk and Gurvich, *Zavod. Lab.*, 1951, **3**, 364), but when using tetramethyl- or tetraethyl-ammonium hydroxide the preparation is somewhat simplified. No interference is caused by the presence of up to 5% of Mg^{2+} and Al^{3+}; the presence of $\simeq$ 0·5% of TiO_2 or CaO causes interference. The range and standard deviation of the results of the determination are, respectively, 0·0007 and $\pm$ 0·03. C. D. KOPKIN

3609. **Complexometric determination of sodium, potassium, or the sum of these, by ion exchange.** E. Gagliardi and H. Reimers (Univ. Graz, Austria). *Z. anal. Chem.*, 1958, **160** (1), 1–6.—Quant. exchange of Na^+ and K^+ for Mg^{2+} can be achieved by passing a soln. of their salts at 5 ml per min. through a column (20 mm $\times$ 250 mm) of ion-exchange resin (Lewatit S100) previously treated with 5% $MgCl_2$ soln. The resulting magnesium soln. can be titrated with EDTA, with Eriochrome black T as indicator, at 80° and pH 9 (NH_4Cl - aq. NH_3 buffer). The water used to remove excess of $MgCl_2$ in the pre-treatment and for the final washing of the column must be free from CO_2. In soln. containing both Na and K the K can first be determined gravimetrically by means of Na tetraphenylboron and the Na is obtained by difference. The method is rapid and accurate. G. BURGER

3610. **Quantitative determination of potassium with sodium tetraphenylboron.** W. Lieber. *Zement-Kalk-Gips*, 1957, **10** (1), 61–62.—A precipitation - titration procedure is described. The sample is dissolved by an appropriate method, filtered, neutralised with NaOH soln., and diluted to contain about 0·25 g of K_2O per 100 ml of soln. To 100 ml of this soln. is added 1 ml of conc. HCl, with thorough mixing, then 20 ml of precipitant soln. (1·2 g of Na tetraphenylboron in 100 ml of water) is added and the combined soln. are stirred frequently for 5 to 10 min. The ppt. is collected in an A2 porcelain filter, and washed with 0·12% Na tetraphenylboron soln. (2 $\times$ 5 ml) and with water (3 $\times$ 5 ml), then dissolved in 10 ml of acetone, the resulting soln. being sucked through the filter into 10 ml of satd. aq. $HgCl_2$ soln. An excess of 0·1 *N* NaOH (20 ml) is added and the soln. is heated to just below the b.p.; 10 ml of 10% aq. KI soln. and 0·5 ml of methyl red - bromocresol green mixed indicator are added and the hot soln. is titrated with 0·1 *N* HCl to the red end-point. The soln. is then treated with 1 to 2 ml of 10% aq. KI soln. and with exactly 3 ml of 0·1 *N* HCl, heated to boiling-point, and titrated with 0·1 *N* NaOH to the green end-point (the KI prevents the hydrolysis of excess of $HgCl_2$ by complex formation). Interference is caused by Rb^+, Cs^+, NH_4^+ and Tl^+, but these are not normally present in cement clinker for which the method is particularly intended. (For English translation see U.K.A.E.A. Rep. IGRL-T/W-69, 1958.) A. JOBLING

3611. **Volumetric determination of potassium after precipitation as tetraphenylboron.** N. Mével and B. Lacruche (Soc. d'Études Chim. pour l'Ind. et l'Agric., 98 bis, Boulevard Héloise, Argenteuil, France). *Mikrochim. Acta*, 1958, (2), 241–247 (in French).—A volumetric method is recommended in place of the usual gravimetric procedure because the latter often gives high results owing to the presence of decomposition products of the reagent. *Procedure*—To the test soln. (neutral or faintly acid) heated to 70° add the required amount of Na tetraphenylboron ($\simeq$ 3% in H_2O with 4 drops of 0·1 *M* $AlCl_3$ per 100 ml). Filter, and dissolve the ppt. on the filter in acetone. Add an excess of $AgNO_3$ soln. (0·1 or 0·05 *N*) followed by H_2O and HNO_3 until the mixture is just acid. Then add ferric alum indicator and titrate with thiocyanate according to the Volhard method. A procedure for recovering the reagent is included.

D. F. PHILLIPS

3612. **The reaction of hexanitrodiphenylamine (dipicrylamine) with caesium, rubidium, potassium and ammonium.** V. Kouřím, J. Krtil and C. Konecny (Inst. Nucl. Phys., Acad. Sci., Prague). *Chem. Listy*, 1958, **52** (2), 262–268.—The pptn. of alkali metals (particularly Cs) with dipicrylamine has been studied and a procedure for the gravimetric and colorimetric determination of Cs is suggested. With the use of colorimetric and radiometric methods, the solubility of dipicrylaminates of Cs, Rb, K and NH_4^+ in water and in diethyl ether was determined, and from the absorption spectra in the u.v. and visible ranges the behaviour and the structure of these compounds were explained. *Procedure for the gravimetric determination of Cs*—Dissolve the sample (13·1 to 123·1 mg of Cs) in H_2O

(20 ml), heat to between 60° and 70° and add a 100% excess of a filtered soln. of dipicrylamine (3%) in *N* Li_2CO_3. Cool for 3 to 5 hr. at 0° to 1°, filter (glass filter S3), wash the ppt. with anhyd. cooled diethyl ether (10 ml) and dry for 2 hr. at 85° to 90° to constant weight. For the detection of alkali metals a paper impregnated with Li dipicrylaminate was used. The spot was revealed with 0·1 *N* HNO_3. As little as 2 to 5 μg of K, Rb or Cs or 200 μg of NH_4^+ can be detected. Rubidium yields a carmine-red spot, whilst those due to Cs, K and NH_4^+ are orange. J. ZÝKA

3613. **New micro-chemical reaction for copper ion.** V. N. Podchaĭnova and A. G. Mishina. *Trudy Ural'sk. Politekh. Inst.*, 1957, (69), 113–118; *Ref. Zhur., Khim.*, 1958, Abstr. No. 21,149.—A new micro-chemical reaction of Cu^{2+} with thiosemicarbazide (**I**) is studied. The reaction of Cu^{2+} with **I** in neutral or acid media gives a fine cryst. dark-blue ppt. (10^{-2} to 10^{-3} g of Cu per ml) or a dark-blue colour (> 10^{-4} g of Cu per ml). The separation of the ppt. is accelerated by the addition of solid K_2SO_4. The complex contains Cu and S in the ratio 1:3; the probable structure is given. The complex is readily soluble in hot water, and is less so in cold water, in acids and in organic solvents (benzene, toluene, di*iso*amyl ether, dichloroethane, etc.). C. D. KOPKIN

3614. **Application of paper chromatography to the detection of copper in solutions and in biological materials.** I. Krzeczkowska. *Ann. Univ. M. Curie-Skłodowska, D*, 1957, **11**, 199–232.—A new "conical" method of paper chromatography has been developed which concentrates the soln. up to 200-fold. Whatman No. 31 filter-paper is cut into a half circle with a long strip of paper left in the middle. The paper is sewn into a cone, placed in a Petri dish containing the test soln. and covered with a glass funnel in such a way that the strip is inside the funnel stem. Collidine - 0·4 *N* HNO_3 (1:1) or pyridine - 0·5 *N* HNO_3 (1:1) is used as solvent for the copper chromatograms. The paper is dried at room temp. Gaseous forms of developers are preferred. Copper was concentrated by repeated chromatography on the same paper, increasing the surface of the cone, or decreasing the diameter of the strip. The method gives well-formed boundaries and enables Cu to be separated from other elements. It was used to determine Cu in urine, blood and human milk. CHEM. ABSTR.

3615. **Reaction of the cuprous ion with ammonium tetrathiocyanatodiamminochromium.** V. N. Podchaĭnova. *Trudy Ural'sk. Politekh. Inst.*, 1957, (69), 107–112; *Ref. Zhur., Khim.*, 1958, Abstr. No. 21,150.—The method described earlier for the detection and determination of Cu^+ with $NH_4[Cr(SCN)_4(NH_3)_2]$ (**I**) (*cf. Z. anorg. Chem.*, 1939, **225**, 386) is modified by the use of organic reducing agents (hydrazine dihydrochloride, hydroxylamine hydrochloride) instead of K_2SnCl_4 to reduce Cu^{2+} to Cu^+. The limiting sensitivity of the detection of Cu with **I** is 2·5 × 10^{-6} g per ml. The detection of Cu with **I** in ferrous and non-ferrous metals and their alloys is also described. C. D. KOPKIN

3616. **Determination of copper by means of lead diethyldithiocarbamate.** S. E. Kreĭmer and L. P. Butylkin ('Severonikel' Combine). *Zavod. Lab.*, 1958, **24** (2), 131–133.—The only ions capable of displacing Pb from a soln. in $CHCl_3$ of Pb diethyldithiocarbamate are Cu, Hg and Ag, and of these, only Cu gives a coloured complex. The complex with Pb, being colourless, can therefore be considered a specific reagent for Cu. To determine Cu in metallic nickel, the sample (2·5 g) is dissolved in dil. HNO_3 (3:2) and the soln., after evaporation to a small volume, is diluted to 200 ml. An aliquot (10 ml) is shaken twice, or thrice if necessary, with 5-ml portions of the reagent soln. [prepared by treating an aq. soln. of 0·1 g of Pb acetate with 5 ml of 10% KNO_3 soln., 5 ml of *N* KCN (to mask traces of Cu and Fe) and an aq. soln. of 0·1 g of Na diethyldithiocarbamate, dissolving the white ppt. in 250 ml of $CHCl_3$, rejecting the aq. layer, filtering the soln. through a dry filter and diluting to 500 ml with $CHCl_3$]. The aq. soln. is finally extracted with 5 ml of $CHCl_3$, the combined extracts are diluted to 25 ml and the extinction is measured. A blank is carried out at the same time. The soln. that is extracted should have a pH of 3 to 4. With metallic cobalt, evaporation of the soln. in HNO_3 may not remove sufficient acid and it is recommended that the aliquot subsequently obtained be treated with aq. NH_3 and tested with universal indicator paper. In the presence of much Fe the sample soln. is treated with aq. NH_3 to give a ppt., which is then dissolved by careful addition of HNO_3. The method is applied to the determination of small amounts of Cu, 0·005 to 0·2%, in metallic nickel and cobalt, chrome - nickel steel, plain carbon steel, and aluminium. G. S. SMITH

3617. **The use of 5-hydroxy-4-azaphenanthrene for the quantitative determination of copper.** D. Jakubke (Martin Luther Univ., Halle-Wittenberg, Germany). *Z. anal. Chem.*, 1958, **160** (1), 6–10.—Copper is pptd. from slightly alkaline aq. soln. containing Na tartrate by dropwise addition, in the cold with constant stirring, of a 2 to 4% soln. of 5-hydroxy-4-azaphenanthrene. After 2 hr. the soln. is heated to 60° and filtered. The ppt. is dried for 2 hr. at 110° and weighed as $(C_{12}H_8ON)_2Cu$. The precision compares favourably with that obtained by pptn. with oxine. There is no interference from Cr^{3+}, Fe^{3+}, Al^{3+}, Bi^{3+}, Pb^{2+} or Zn^{2+}.
G. BURGER

3618. **New method of analysis of copper alloys.** M. Kuhn. *Chim. Anal.*, 1958, **40** (2), 50–54.—The precision and accuracy of the C.T.I.F. (Centre Technique des Industries de la Fonderie) method of analysis of copper alloys are evaluated and compared with those of other methods.
A. R. ROGERS

3619. **Determination of copper and arsenic in carbon blacks.** R. R. Gibbs and F. Moore (R. W. Greeff & Co. Ltd., London). *Rubb. J.*, 1958, **134** (14), 524–525.—In the determination of Cu, the carbon (15 to 20 g) is ignited at 500° to 550°, and the residue is dissolved in HNO_3. Copper is converted into Cu diethyldithiocarbamate, which is extracted with CCl_4 and determined colorimetrically or photometrically. In the determination of As, carbon black is treated with brominated HCl (1 ml of Br in 100 ml of conc. HCl) and heated in a stream of HCl. Arsenic is distilled off as the trichloride and determined in the distillate by the Gutzeit disc method. O. M. WHITTON

3620. **Colorimetric determination of copper in aluminium, cadmium, zinc and nickel with *p*-anisidine and potassium thiocyanate.** V. N. Podchaĭnova. *Trudy Ural'sk. Politekh. Inst.*, 1957, (69), 119–125; *Ref. Zhur., Khim.*, 1958, Abstr.

No. 21,151.—The reaction of Cu^{2+} with *p*-anisidine (*Ref. Zhur., Khim.*, 1954, Abstr. No. 43,472) in the presence of SCN^- is used for the determination of Cu. To determine Cu in aluminium, dissolve the sample (2 g) in a mixture of conc. HCl and conc. HNO_3 (15 to 20 ml) and evaporate almost to dryness. To the residue add conc. HCl (0·2 or 0·3 ml) and water (10 ml) and filter. To the soln. add conc. aq. NH_3 to a slight cloudiness, then remove this with conc. HCl. Heat the soln. to boiling, add a 20% soln. of $Na_2S_2O_3$ (two or three drops) to reduce Fe^{3+}, and a suspension of CdS (1 or 2 ml) (≡ 0·02 to 0·04 g of the dry powder); mix, heat to boiling, filter and wash the ppt. with hot 10% HCl till the CdS is completely dissolved. Dissolve the CuS on the filter with hot conc. HCl and wash with water. Combine the filtrate and wash liquors and dilute to 25 or 50 ml. To 15 or 20 ml add conc. aq. NH_3 (1 ml), 2 *N* acetic acid (2 or 3 ml) and the reagent (a mixture of equal vol. of 2 *N* KSCN and 0·2 *N* *p*-anisidine) (1 or 2 ml), make up to 50 ml and after 15 or 20 min. measure the extinction of the red - violet soln. The determination of Cu in nickel, cadmium and zinc is carried out similarly. The error for Cu in aluminium and nickel is ≃ 0·001% and in cadmium and zinc from − 0·0002 to + 0·0001%. The determination takes 40 to 45 min. C. D. KOPKIN

3621. **Photometric determination of copper in metallic tin by the diethyldithiocarbamate method.** Sasiti Ikeda and Hiroshi Nagai (Saganoseki Smelting Plant, Nippon Mining Co., Ooita-ken). *Japan Analyst*, 1958, **7** (2), 76–79.—Most of the Sn in metallic tin is separated by pptn. as oxalate, any Cu being masked with EDTA. Since EDTA retards the extraction with diethyldithiocarbamate (**I**) in CCl_4, the Cu is demasked with Fe^{3+}. The time taken for a determination is < 60 min. and the error is < 5%. *Procedure*—Dissolve the sample (3 to 5 g) in conc. HCl (20 ml), dilute to 130 ml, add EDTA soln. (0·25 *M*, 15 ml) and satd. oxalic acid soln. (40 ml) and filter. Add iron alum soln. (1%, 3 ml) and ammonium citrate soln. (10%, 10 ml) to the filtrate, make ammoniacal, add a soln. of the sodium salt of **I** (0·1%, 2 ml), and shake with CCl_4 (10 ml). Measure the extinction at 440 mμ. K. SAITO

3622. **Polarographic determination of copper, lead and zinc in the presence of ferric iron. I.** D. B. Labkovskaya and L. S. Reishakhrit. *Uch. Zap. LGU*, 1957, (211), 116–122; *Ref. Zhur., Khim.*, 1958, Abstr. No. 842.—The possibility is indicated of determining Cu^{2+}, Pb^{2+} and Zn^{2+} together in the presence of an excess of Fe^{3+} in a basal soln. of 0·1 *N* KCl and 0·03 *N* HCl after reduction of Fe^{3+} to Fe^{2+} with hydroxylamine. Under these conditions, Cu^{2+} are not reduced by hydroxylamine. C. D. KOPKIN

3623. **Analysis for industry [colorimetric determination of silver. II].** R. Goulden. *Ind. Chem. Mfr*, 1958, **34**, 200–202.—A review. (26 references.) O. M. WHITTON

3624. **S-Substituted thioureas as analytical reagents. II. S-Methylthiuronium sulphate as a reagent for silver.** Sushil K. Siddhanta and Satyendra N. Banerjee (Appl. Chem. Dept., Indian Inst. Technol., Kharagpur). *J. Indian Chem. Soc.*, 1958, **35** (1), 53–56.—A highly insoluble, stable ppt. of Ag methylmercaptide ($AgCH_3S$), which coagulates rapidly, is filtered easily and may be dried at 110° to 125°, is produced quant. when aq. NH_3 is added to a mixture of S-methylthiuronium sulphate and $AgNO_3$ in aq. soln. A quant. separation of Ag and Zn is also possible in strongly ammoniacal soln. containing NH_4NO_3 since the Zn methylmercaptide [$Zn(CH_3S)_2$] is not pptd. under these conditions. The $AgCH_3S$ is less soluble than AgCl. I. JONES

3625. **Gravimetric determination of silver as silver chromate.** G. P. Pendse, H. D. Bhargava and B. R. Sant (Victoria College, Gwalior, India). *Z. anal. Chem.*, 1958, **160** (3), 188–189 (in English).—The soln. of Ag, as nitrate, is poured slowly with stirring into an excess of aq. K_2CrO_4. The brownish-red ppt. is filtered off, washed with cold H_2O and dissolved in aq. NH_3. On boiling off the NH_3, greenish-black $AgCrO_4$ is pptd. The ppt. is collected on the same filter and either washed with cold H_2O and dried at 110° to 120°, or washed successively with H_2O, ethanol and diethyl ether and dried at 50° to 60°. The results of 6 determinations on 0·05 to 0·66 g of Ag_2CrO_4 lie within 1·9 mg of the theoretical. G. BURGER

3626. **Radiochemical determination of gold and the platinum metals in refined silver and cathode nickel.** O. E. Zvyagintsev and A. I. Kulak. *Zhur. Neorg. Khim.*, 1957, **2** (7), 1687–1692; *Ref. Zhur., Khim.*, 1958, Abstr. No. 7644.—The radiochemical determination of Au, Pt, Pd and Ir in refined silver and cathode nickel is carried out after their chemical separation with the use of isotopic carriers. The irradiation of Au, Pt, Pd and Ir with slow neutrons gives, by the (n, γ) reaction, radioactive isotopes of these elements, with half-lives that allow a chemical removal and separation of the elements in a radiochemically pure state, and which also allows radiometric measurements to be carried out. C. D. KOPKIN

3627. **Determination of a minute amount of magnesium in brine.** Masami Suzuki, Hiroshi Kondo and Shizo Hirano (Fac. of Engng, Nagoya Univ., Chikusa-ku). *Japan Analyst*, 1958, **7** (1), 47–49.—Since NaCl (> 2%) interferes with the colorimetric determination of Mg (< 100 μg) with Titan yellow (**I**), Mg is co-pptd. with Cu (≃ 5 mg); co-pptd. Hg is masked with KCN. Interference results from a large amount of Fe and Al. The sample (25 ml) is boiled with $CuCl_2$ soln. (5 mg per ml, 1 ml) and NaOH soln. (20%, 1 ml); the ppt. is washed with 0·01 *N* NaOH, then dissolved in warm HCl (0·3 *N*, 5 ml) and treated with hydroxylamine hydrochloride soln. (5%, 1 ml), $CaCl_2$ soln. (1%, 2 ml), poly(vinyl alcohol) (1%, 10 ml), KCN soln. (1%, 3 ml), aq. **I** soln. (0·2%, 1 ml) and NaOH soln. (20%, 5 ml) (total vol. 100 ml). Photometric measurement is made at 532 mμ after 10 min. K. SAITO

3628. **Determination of magnesium in the presence of aluminium.** M. V. Liberman. *Zavod. Lab.*, 1958, **24** (2), 147.—Improvements in the method of Pribil (*Anal. Abstr.*, 1955, **2**, 309) and Ritchie (*Anal. Abstr.*, 1955, **2**, 2354) are described. The triethanolamine used must be very pale in colour. Darker soln. should be re-distilled (20 mm of Hg) and the fraction from 170° to 225° collected. Titration should be carried out at between 4° and 5° (*cf.* Ritchie). The sample soln. (10 ml), *e.g.*, that obtained during the analysis of Al - Mg alloys containing 10 to 70% of Al, is mixed with 250 ml of water, 0·5 g of NH_4Cl and 5 ml of triethanolamine,

and then with 5 ml of a buffer soln., prepared by dissolving 54 g of NH_4Cl in 350 ml of 25% aq. NH_3 and diluting to 1 litre, and 5 drops of Eriochrome black T indicator soln. The soln. is cooled in melting ice and titrated with 0·1 *N* EDTA (disodium salt) until the colour changes from reddish violet to blue. G. S. SMITH

3629. **Rapid method of determining magnesium in magnesium - ferrosilicon alloy.** A. V. Evko. *Zavod. Lab.*, 1958, **24** (2), 147–148.—If the alloy (0·2 to 0·5 g) is boiled for 15 min. with 60 ml of water and 12 ml of conc. HCl, the whole of the Mg dissolves and can be determined by the volumetric 8-hydroxyquinoline method after removal of dissolved sesquioxides. G. S. SMITH

3630. **The analysis of dolomitic rocks by simultaneous thermogravimetric determination of calcium and magnesium.** T. Dupuis and J. Dupuis (11, rue Pierre Curie, Paris). *Mikrochim. Acta*, 1958, (2), 186–196 (in French).—A simple, rapid and more accurate method than the complexometric one is described. It is generally applicable and can be used even if a thermobalance is not available. *Procedure*—Place the crucible containing the carbonate ppt. in a furnace which can be regulated. Heat it first at $\simeq 500°$ (to give MgO plus $CaCO_3$) and then at $\simeq 900°$ (to give MgO plus CaO) until constant weight is attained. Make several successive weighings to ensure that constant weight at 500° has been attained, and keep the crucible at 500° to 550° for about 30 min. while recording the thermolysis curve in order to establish a plateau. The method may be used to determine Ca and Mg present in ratios other than those in dolomites. D. F. PHILLIPS

3631. **The determination of calcium, with Eriochrome red B as indicator.** L. Jankovits and L. Erdey (Tech. Univ., Budapest). *Acta Chim. Acad. Sci. Hung.*, 1958, **15** (2), 163–174 (in German).—A 0·06% aq. soln. of Eriochrome red B (0·5 ml) is a suitable indicator for the complexometric titration of Ca^{2+} in soln. buffered at pH 9 to 10. The end-point is indicated by a change from yellow to red and can be determined within 2 to 3 drops of 0·01 *M* soln. or 1 to 2 drops of 0·05 *M* soln. Interference is caused by Ba, Sr, Mg, Fe, Co, Ni, Cr, Al, Zn, Cu, Pb, Cd, Mn and Bi. [*Cf. Magyar Kém. Foly.*, 1958, **64**, 50 (in Hungarian).] G. BURGER

3632. **Complexometric titrations (chelatometry). XXXVI. The screening of iron by triethanolamine: the determination of calcium with thymolphthalein complexone.** R. Přibil, J. Körbl, B. Kysil and J. Vobora (Lab. Anal. Chem., Acad. Sci., Prague). *Chem. Listy*, 1958, **52** (2), 243–246.—Large amounts of Fe (5 to 10 mg) cannot, for the most part, be readily masked with triethanolamine (**I**). This disadvantage can be avoided if, before the addition of **I**, a suitable amount of EDTA (disodium salt) (**II**) (equiv. to about one-third of the Fe) is added. The resulting soln. in NaOH medium is then colourless. This procedure can be used for the determination of Ca in the presence of Fe; a known amount of **II** is added and its excess is determined by titration with $CaCl_2$ soln. (thymolphthalein complexone as indicator). *Procedure for Ca in calcium silicate*—Mix the sample (1 g) with Na_2CO_3 - K_2CO_3 (1 : 1) (2 g) and heat in a silver crucible; add Na_2O_2 (5 g) with stirring and fuse. Cool the melt, wash it with H_2O (100 ml) and boil (3 min.). Make the soln. slightly acid with HNO_3, boil for 1 min. and precipitate the Ag^+ with NaCl. Cool to 60° and add gelatin (1% soln.) (5 drops) for pptg. SiO_2, and mix. Cool, transfer to a 250-ml flask, make up to vol. with H_2O, mix and filter. To a 100-ml portion of the filtrate add 0·1 *M* **II** (40 ml) and **I** (1 : 2) (10 ml) and add 2 *N* NaOH till the soln. is colourless. Add indicator and titrate with 0·05 *M* $CaCl_2$ till blue. The accuracy is $\pm$ 0·15%; the time required is 50 min. J. ZÝKA

3633. **Determination of the alkaline-earth metals (barium, calcium, magnesium) by homogeneous precipitation.** K. I. Litvin. *Sb. Nauch. Rabot, Moskov. Gorn. Inst.*, 1957, (1), 97–105; *Ref. Zhur., Khim.*, 1958, Abstr. No. 854.—The determination of Ca with dimethyl oxalate (**I**) (Gordon and Wroczynski, *Anal. Chem.*, 1952, **24**, 896) has been improved. To 50 ml of a boiling soln. containing 20 to 125 mg of Ca add 10 ml of a 4% aq. soln. of **I** at 60° to 70°, and aq. NH_3 dropwise till the soln. is neutral, then boil it for 2 or 3 min. and set it aside for 10 min. in the cold. Filter off the Ca oxalate and determine the Ca in the usual way. To determine Ba and Ca when present together, mask the Ca by adding glycerol or sucrose, and precipitate Ba with dimethyl sulphate; precipitate Ca in the filtrate with **I**. For the pptn. of Mg, use is made of $POCl_3$, which hydrolyses to form PO_4^{3-}. In this way it is possible to determine Mg in the filtrate after pptg. Ba with dimethyl sulphate and Ca with **I**. The error when determining Ba, Ca and Mg in the presence of each other is $\pm$ 3·6% for each of the elements. C. D. KOPKIN

3634. **Flame-photometric determination of calcium, strontium and barium in the presence of each other.** W. Schuhknecht and H. Schinkel (Ensheimer Str. 35, St. Ingbert, Saar). *Z. anal. Chem.*, 1958, **160** (1), 23–33.—After being separated from heavy metals, SiO_2, PO_4^{3-} and alkali metals, Ca, Sr and Ba can be determined in a filter-type flame photometer. Mutual interference is eliminated by the procedure described. The appropriate wavebands are, for Ca, 622 mμ when Ba predominates, and 553·3 mμ when Sr predominates; for Sr, 671 mμ or, with much Ca present, 460·7 mμ; for Ba, 873 mμ. For each metal the effect of a known addition of each of the other two is determined for the apparatus in use. These results are used to correct the Ca, Sr and Ba readings obtained with the sample to give approx. values for the concn. For each metal two soln. are then prepared such that the concn. of the metal to be determined is bracketed and those of the other two are equal to the approx. value found. By interpolation, each alkaline-earth metal can be determined within 3% when its concn. ratio with the other two is 1 : 100, and within 10% when the concn. ratio is 1 : 1000. G. BURGER

3635. **Separation and determination of small amounts of barium in the presence of strontium and calcium.** A. Tockstein and V. Novák (Inst. Anal. Chem., High School Chem. Technol., Pardubice, Czechoslovakia). *Chem. Listy*, 1958, **52** (2), 269–275.—When a cobaltous salt is added to a cooled ammoniacal soln. of a mixture of salts of Ca, Sr and Ba containing EDTA (disodium salt) (**I**) and sulphates, only $BaSO_4$ is pptd. owing to the demasking action of Co^{2+}. The $BaSO_4$ can be separated by centrifugation and determined polarographically by the method of Tockstein and Šerák (*Chem. Listy*, 1952, **46**, 539). *Procedure*—

To a mixture of the nitrates of Ca, Sr and Ba (in a centrifuge tube) add a very small excess of 0·1 to 0·2 *M* **I** (when necessary, first determine complexometrically the total amount of Ca, Sr and Ba). Add a satd. soln. of K_2SO_4 in aq. NH_3 (5 ml) and 3 to 5 ml of 11 *M* aq. NH_3. Close the tube and cool for 10 min. in ice. Stir and add a small excess of 0·1 *M* to *M* $Co(NO_3)_2$; $BaSO_4$ is pptd. Cool with ice (2 to 3 min.) and centrifuge (10 min.). Add acetone (2 drops) and carefully separate the supernatant liquid. To the ppt. add 0·1 *M* aq. ammoniacal **I** (0·05 to 0·1 ml) and dissolve with gentle warming. Repeat the pptn. Separate the liquid as before and add to the ppt. a known amount of aq. ammoniacal soln. of the complex of Ag with EDTA (1 to 2 ml of 0·005 *M* soln.). When the ppt. is dissolved, add gelatin (0·5%) (0·5 ml) and fuchsine (satd. soln. diluted 1:10) (0·25 ml) and dilute to 10 ml. Determine polarographically the height of the wave of the silver complex. Make a blank determination. A mathematical explanation of the principle of the determination and a statistical evaluation of errors are given. J. ZÝKA

3636. **Determination of zinc in iron ores in the presence of cobalt.** Yoshimi Umezaki (Resources Res. Inst., Kotobuki-cho, Kawaguchi, Saitama-ken). *Japan Analyst*, 1958, **7** (1), 37–42.—The ion-exchange separation of Zn from Co and Fe (*cf.* Kraus and Moore, *J. Amer. Chem. Soc.*, 1953, **75**, 1460) was applied to the analysis of iron sulphide ores and related intermediates. When the column of Dowex 1-X8 is eluted with 2 *N* HCl, all the Co (< 20 mg) and most of the Fe (< 400 mg) are washed from the resin phase, but Zn (< 100 mg) remains adsorbed. The Zn is eluted with 0·5 *N* HNO_3 (80 ml) and determined polarographically with pyridine as supporting electrolyte in a soln. containing < 10 mg of Zn per 100 ml. *Procedure*—Decompose the sample (1 g) with HCl and H_2O_2 or HNO_3, evaporate to dryness and dissolve the residue in HCl (2 *N*, 10 ml). Pass this soln. through the column (diameter 9 mm) of Dowex 1-X8 (50 to 100 mesh, ≃ 10 ml) and elute first with 2 *N* HCl (2·5 ml per min., 80 ml) and then with 0·5 *N* HNO_3 (80 ml). Evaporate the HNO_3 soln., neutralise it with NaOH, make acid with HCl (4 *N*, 6 ml), boil with pyridine (50% aq. soln., 10 ml) and make up to 100 ml. Filter the product and measure the polarographic currents at − 0·80 to 1·2 V *vs.* the S.C.E.

K. SAITO

3637. **Analytical applications of Dutoit's thermovolumetry. II. Analysis of zinc in brass.** Kumar K. Chatterji (Phys. Chem. Dept., Univ. Coll. Sci. and Technol., Calcutta). *J. Indian Chem. Soc.*, 1958, **35** (1), 57–62.—The thermometric method of Dutoit (*J. Chem. Phys.*, 1922, **19**, 324, 331) is used for the determination of Cu plus Zn by titration with $(NH_4)_2Hg(SCN)_4$. In the presence of Cu and in brass, Zn is determined by the same reagent after removal of Cu as $Cu_2(SCN)_2$ by the addition of NH_4SCN and H_2SO_3. The results for Zn are usually ≃ 1% higher than those obtained by gravimetric estimation as NH_4ZnPO_4.

I. JONES

3638. **Application of complexones to the rapid determination of zinc in brass.** C. Pépin-Donat. *Chim. Anal.*, 1958, **40** (2), 47–49.—Procedures are given for the rapid determination of Zn, and for Pb and Mn when these metals are present. *Zinc (lead and manganese absent)*—Heat the sample (0·25 g) with HNO_3 (5 ml) until fuming ceases, cool, add H_2O (100 ml), neutralise with aq. NH_3 and add an excess (10 ml). Decolorise with 20% KCN soln. and add 5 ml in excess. Dilute to 300 ml, add 0·4% ethanolic Eriochrome black T soln. (**I**) (1 ml) and 4% aq. formaldehyde soln. (20 ml) and titrate immediately with 0·05 *N* EDTA (disodium salt) (**II**) to a blue colour. The precision is ≃ ± 0·6%. The interference of Al is masked by addition of KF and that of Fe by triethanolamine; Sn does not interfere. *Zinc and lead*—Dissolve 0·25 g of sample in HNO_3 (5 ml), remove nitrous fumes, add H_2O (100 ml) and mannitol (1 g), neutralise with aq. NH_3 and add 10 ml in excess. Decolorise with 20% aq. KCN soln. and add 5 ml in excess. Add 5 ml of a soln. obtained by mixing a $MgCl_2$ soln. containing 5 mg of Mg in 50 ml with aq. NH_3 - NH_4Cl buffer of pH 10 (10 ml), heating to 50° and titrating with 0·005 *N* **II**. Titrate the Pb with 0·005 *N* **II**, with **I** as indicator. Then dilute to 300 ml, add 4% aq. formaldehyde soln. (20 ml) and titrate the Zn as described above. *Zinc and manganese*—Dissolve 0·25 g of sample as described above, add H_2O (100 ml) and ascorbic acid (0·2 g), neutralise with aq. NH_3 and add 10 ml in excess. Decolorise with 20% aq. KCN soln. and add 5 ml in excess. Add 30% aq. triethanolamine (15 ml), heat almost to boiling and titrate the Mn and Pb in the hot soln. with 0·005 *N* **II**, with **I** as indicator. Cool, dilute to 300 ml, add 4% aq. formaldehyde soln. (20 ml) and titrate the Zn as described above.

A. R. ROGERS

3639. **Polarographic determination of zinc and manganese in the presence of ferrous iron. II.** L. S. Reishakhrit and A. M. Naumova. *Uch. Zap. LGU*, 1957, (211), 123–128; *Ref. Zhur., Khim.*, 1958, Abstr. No. 843.—The possibility is indicated of determining Zn^{2+} and Mn^{2+} together in a basal soln. of *N* KCl. With ratios of Zn to Mn of 1:1, 1:2, 1:5, 1:10, 1:44, 2:1, 6:1 and 11:1, there is a straight-line dependence of the value of the limiting current *I* on the concn. of the corresponding ion, the coeff. of proportionality between *I* and the concn. of one ion in the presence of the other decreasing approximately by half. The value of *I* is almost independent of the acidity of the soln. in the neutral and weakly acid region, up to a pH of < 3·5 for Zn^{2+} and a pH of < 4·25 for Mn^{2+}. The value of *I* for Zn^{2+} is directly proportional to the concn. of Zn^{2+} even in the presence of Fe^{2+}; in determining Mn^{2+} in the presence of Fe^{2+} it is necessary that the concn. of Fe^{2+} be constant, or that the Fe^{2+} be complexed. C. D. KOPKIN

3640. **Gravimetric micro-determination of cadmium with 2-*o*-hydroxyphenylbenzoxazole.** C. Comerman and R. Pavlovec (Israel Inst. of Tech., Haifa). *Mikrochim. Acta*, 1958, (2), 259–265 (in English).—2-*o*-Hydroxyphenylbenzoxazole reacts with Cd^{2+} in alkaline soln. to form a yellow ppt. which, after being dried, corresponds to $Cd(C_{13}H_9O_2N)_2$. After a systematic study of all the factors affecting this determination, an exact gravimetric micro-method has been developed for the determination of Cd, use being made of Emich's micro-technique and a Pyrex-glass filter-stick. The max. relative error is ± 0·6%.

D. F. PHILLIPS

3641. **Volumetric determination of mercury(I) with permanganate.** G. Jagga Rao, K. Bhaskara Rao and G. Gopala Rao (Andhra Univ., Waltair, India). *Z. anal. Chem.*, 1958, **160** (2), 115–117 (in English).—In HCl soln. of pH > 3, Hg^+ can be

titrated directly with $KMnO_4$ in the presence of ICl. An aliquot of the sample soln. with 0·02 *M* ICl (5 ml) is made up to 50 ml with H_2O and HCl, and CCl_4 (5 ml) is added. The soln. is titrated with $KMnO_4$ until the colour in the CCl_4 is discharged. The end-point is sharp and reversible. Sulphate must not be present in concn. $> N$.

G. BURGER

3642. **Colorimetric determination of boron.** M. Beck (Inst. for Inorg. and Anal. Chem., Sci. Univ. of Szeged, Hungary). *Magyar Kém. Foly.*, 1958, **64** (2), 61.—It has been claimed (*cf.* Monnier *et al.*, *Anal. Chim. Acta*, 1947, **1**, 13) that B can be determined by the following sequence of reactions—the Fe^{3+} - sulphosalicylic acid complex (**I**) is partially decolorised by the excess of F^- over the amount required to form BF_4^-. Examination of the method showed that the B content is only partly responsible for the change in extinction, because the change in pH (which is governed by the buffering capacity of the soln.) also influences the extinction. The indicator properties of **I** also interfere. When the pH was kept constant, the change in extinction was insufficient to form the basis of a successful method. A. G. PETO

3643. **Determination of a small amount of boron by coulometric titration. I. Acid - base titration in the presence of mannitol.** Hiroshi Iinuma, Takayoshi Yoshimori and Hironori Takeuchi (Fac. of Engng, Gifu Univ., Gifu). *Japan Analyst*, 1958, **7** (1), 8–11.—The coulometric titration of H_3BO_3 (< 50 micro-equiv.) is carried out by the use of a large amount of mannitol (3·5 g for 50 ml of electrolytic soln.) in 0·2 *M* NaBr or Na_2SO_4. The sample soln. (or the distillate of methyl borate) is exactly neutralised to a pH of 6·3 to 6·8, mixed with mannitol and submitted to const.-current electrolysis (platinum electrodes) until the pH becomes identical with the initial value. The amount of B is calculated from the duration of electrolysis with an error < 6·5%. K. SAITO

3644. **Determination of boron by the neutron method under field conditions.** V. K. Khristianov, G. I. Panov and A. A. Chernova. *Geokhimiya*, 1957, (2), 95–101; *Ref. Zhur., Khim.*, 1958, Abstr. No. 7588.—A method is described for the determination of B in B-containing rocks with an instrument that has been constructed in two forms, for use in the laboratory and in the field. The method is based on the high effective cross-section for the capture of thermal neutrons by B, which is several orders greater than that of the nuclei of the main rock-forming elements. On bombarding the rock with neutrons there is observed a weakening of the neutron flow, which is proportional to the B content of the rock. In determining B by this method, which comprises a measurement with the B-containing rock as compared with one with a similar sample not containing B, no preparatory chemical treatment of the samples is required. The error due to the difference in composition of standards and sample is $\pm$ 0·15% of B_2O_3, and with a satisfactory choice of samples the error decreases to 0·03% of B_2O_3. The reproducibility of results with > 0·5% of B_2O_3 is $\pm$ 10%. The error of the determination, as compared with chemical methods of analysis, is $\simeq$ 20%. It is possible to carry out 100 analyses in $\simeq$ 8 hr. C. D. KOPKIN

3645. **The decomposition of minerals with aluminium - sodium hydroxide. Determination of boron in silicates.** M. Štefl (Chem. Department, High-School Agric., Prague). *Chem. Listy*, 1958, **52** (2), 342–343.—*Procedure*—To the sample (0·2 g of tourmaline or 0·3 g of glass) in a suitable tube (20 mm × 160 mm) add about 0·1 g of finely powdered aluminium and 0·8 to 1 g of dry powdered NaOH. Mix, and heat the tube slightly, in a horizontal position, with a small flame. After the evolution of H has ceased (2 to 5 sec.), continue heating the tube for 2 to 3 min. Moisten the lower part of the tube with methanol and transfer the contents to a distillation flask, by means of the same solvent. Add H_2SO_4 and distil the methyl borate into K_2CO_3 soln. Evaporate this distillate to dryness, ignite the residue, dissolve in H_2O and determine boron photometrically (with carmine as reagent, at 585 mμ). The results were higher than those obtained by classical methods of decomposition, probably owing to a more complete decomposition with the use of Al - NaOH.

J. ZÝKA

3646. **Volumetric determination of aluminium in clays with 8-hydroxyquinoline without preliminary removal of silicic acid.** N. Isakova and S. Mireva. *Khim. i Ind., Sofia*, 1957, **29** (4), 25–27; *Ref. Zhur., Khim.*, 1958, Abstr. No. 10,982.—Fuse the sample (0·2 g) in a platinum crucible with 2 g of Na_2CO_3 or a mixture of Na_2CO_3 and K_2CO_3. Treat the melt with water, neutralise with HCl, add a few ml of 3% H_2O_2, boil for 10 min., cool, add 20% NaOH soln. (25 ml), boil for several minutes, make up to 250 ml and filter. To 50 ml of the filtrate, acidified to pH 3 or 4, add, at 80° to 90°, 1 ml of 8-hydroxyquinoline soln. (5 g of reagent in 100 ml of 2 *N* acetic acid) for each 3 mg of Al, 2 *N* ammonium acetate till pptn. ceases, and a further 25 ml of the 2 *N* ammonium acetate. After cooling, filter off the ppt., wash it with hot water till a clear colourless filtrate is obtained, and dissolve it in 2 to 2·5 *N* HCl (50 ml). To the soln. obtained add KBr (0·5 g) and titrate with 0·1 *N* $KBrO_3$ in the presence of 5 or 6 drops of a 0·1% soln. of methyl red to a yellow colour. After 2 min. add KI (0·5 g) and titrate the liberated iodine with 0·1 *N* $Na_2S_2O_3$. The quantity of Al_2O_3 is found by the difference of the titrations. The analysis takes $\simeq$ 3 hr. C. D. KOPKIN

3647. **Complexometric determination of aluminium in cryolite-type salts.** K. Grüner, M. Soukup and B. Drahotská. *Hutn. Listy*, 1958, **13** (2), 153–154.—*Procedure*—Evaporate the sample (1 g) in a platinum dish with H_2SO_4 (1:1) (10 ml). Transfer the wet residue with hot water into a 500-ml titration vessel, and dissolve the ppt. with NaOH soln. (30%) added dropwise. Cool the soln. and neutralise with H_2SO_4 (1:1) (methyl orange indicator). Add 15 to 20 ml of 0·1 *M* EDTA (disodium salt), ammoniacal buffer soln. (pH 10) (5 ml) and Eriochrome black T as indicator, and titrate with 0·1 *M* $MgSO_4$ till wine red. The titration must be carried out very rapidly to ensure a sharp indicator change.

J. ZÝKA

3648. **Determination of aluminium and iron in various materials from aluminium works with EDTA (disodium salt).** A. A. Bashkirtseva and E. M. Yakimets. *Trudy Ural'sk. Politekh. Inst.*, 1957, (58), 76–87; *Ref. Zhur., Khim.*, 1958, Abstr. No. 4339.—To determine Al^{3+}, add to the soln. excess of a standard soln. of EDTA (disodium salt) (**I**), heat to between 70° and 80°, add 100 ml of hot water, neutralise, add 10 ml of acetate buffer soln. (pH 6·0), and after 2 or 3 min. titrate the excess of **I** with a 0·025 *M* soln. of Al^{3+} to haematoxylin

Interference is caused by Fe^{3+}, Fe^{2+}, Cu^{2+}, Ti^{4+}, Zn^{2+} and Mn^{2+}, but not by Na^+, K^+, Cl^-, SO_4^{2-}, NO_3^- or CH_3COO^-. In determining Al in aluminate soln. (in the absence of interfering ions), neutralise the hot soln. to phenolphthalein with 2 *N* acetic acid. The determination of Fe is carried out (after oxidation to Fe^{3+}) by direct titration of the hot soln. (pH 1 to 2) with **I** soln. in the presence of SCN^- or Na sulphosalicylate. Interference in the determination of Fe is caused by Cu^{2+}, Zn^{2+}, Ni^{2+}, Co^{3+}, Cr^{3+} and > 10 mg of Al. C. D. KOPKIN

3649. **Spectrophotometric determination of aluminium and iron in industrial silicons.** J. Bril (Compagnie Pechiney, Saint-Jean-de-Maurienne, France). *Mikrochim. Acta*, 1958, (2), 212–219 (in French).—A method for the simultaneous colorimetric determination of Al and Fe with ferron has been perfected and applied to the determination of these elements in silicon and ferrosilicon. The method is based on Davenport's procedure (*cf. Brit. Abstr. C*, 1949, 373). The limit of detection for Al is ≃ 0·02 μg per ml (measured at 365 mμ) and for Fe 0·05 μg per ml (measured at 600 mμ). With ferrosilicon, Fe is extracted with ether before the determination of Al. The accuracy claimed is similar to that of the gravimetric oxinate procedure, but the time required is much shorter.

D. F. PHILLIPS

3650. **Spectrographic determination of gallium in silicate rocks.** Hiroshi Hamaguchi and Kenji Tomura (Fac. of Sci., Tokyo Univ. of Education, Koishikawa). *Japan Analyst*, 1958, **7** (2), 105–106.—Results of the spectrographic determination of Ga (10 to 300 p.p.m.) by a method previously described (Hamaguchi and Kuroda, *Anal. Abstr.*, 1956, **3**, 125) were compared with those obtained by fluorimetric analysis. The agreement is satisfactory.

K. SAITO

3651. **Gallium ferrocyanides and their analytical use.** I. V. Tananaev and N. V. Bausova. *Khimiya Redkikh Elementov*, 1957, (3), 41–56; *Ref. Zhur., Khim.*, 1958, Abstr. No. 7511.—The systems $GaCl_3$ - $M_4[Fe(CN)_6]$ - H_2O, where M is Li, Na or K, have been studied by solubility, light absorption, e.m.f. and electrical conductivity methods. New methods for determining Ga have been evolved, namely potentiometric [with $Na_4Fe(CN)_6$], amperometric and heterometric. It is shown that the first two methods may be used for the determination of small quantities of Ga in the presence of large quantities of Al. C. D. KOPKIN

3652. **Colorimetric determination of indium with di-2-naphthylthiocarbazone.** W. G. Britton (Colorado Univ., U.S.A.). *Dissert. Abstr.*, 1957, **17** (12), 2787.—Buffers containing In were shaken with CCl_4 soln. of di-2-naphthylthiocarbazone (**I**) to determine optimum conditions of extraction. Acetate and succinate buffers give optimum extraction at pH 5, but slight variations in pH result in a marked decrease in extraction; citrate does not extract at this value, but does at 8·0 to 9·5. No extraction is given by $CHCl_3$ - **I** with any of the buffers. Extraction at pH 9 with citrate buffer and CCl_4 - **I** was chosen for the analytical method. Initial pptn. with aq. NH_3 separates In in large amounts from interfering metals; ether extraction removes most of the Fe, while oxine removes In, Cu, Bi and Al from soln. After the metal has been decomposed by $HClO_4$ oxidation, the CCl_4 - **I** reagent extracts In, Cu and Bi. Indium is separated from Cu and Bi by dil. HCl with citrate buffer at pH 9, and the In is again extracted with **I**. The extinction of the organic layer containing the indium complex is read, and the complex is reverted with dil. HCl and a further reading is made. The change in extinction gives the In present. This reversion technique eliminates the necessity for frequent standardisation of **I**. S.C.I. ABSTR.

3653. **The electrolytic determination of indium.** H. Terrey and J. Thabit (University Coll., London). *J. Chem. Soc.*, 1958, 1303.—To avoid attack on the platinum electrode, a known amount of cupric sulphate (≡ ≃ 15 mg of Cu) is added to the indium sulphate soln., which is made alkaline with NaOH or aq. NH_3 soln. Enough formic acid is added to make the soln. acid, discharge the blue colour and re-dissolve the gelatinous ppt. At about 2 V, Cu is deposited first, then the potential is increased to allow a current of 4 to 5 amp. to deposit the In on the electrode. N. E.

3654. **Polarographic determination of indium in zinc concentrates containing a high percentage of iron.** M. Ostanek ("J. Stefan" Inst., Ljubljana, Yugoslavia). *Rep. "J. Stefan" Inst., Ljubljana*, 1957, **4**, 147–149 (in English).—Indium is extracted from zinc concentrates in 6 *M* HBr by shaking twice with di*iso*propyl ether. Iron, which is extracted with the In, is oxidised with HNO_3 and removed by extraction with di*iso*propyl ether from 7 *M* HCl. The polarogram is recorded in HCl soln. (< 2 *M*) containing gelatin. The results are in agreement with those of flame-photometric determinations. No interference is caused by Na, Fe, Bi, Si, Mg, Mn, Pb, B, Ca or Al. G. BURGER

3655. **Chromate method for determining thallium.** N. I. Bashilova. *Khimiya Redkikh Elementov*, 1957, (3), 105–113; *Ref. Zhur., Khim.*, 1958, Abstr. No. 877.—Testing of the chromate method for determining Tl at various concn. of K_2CrO_4 and NH_3 shows that the concn. of K_2CrO_4 necessary to precipitate Tl completely may vary between 1 and 2·5% at a const. concn. of NH_3 of 1%. In the analysis, the Tl should be pptd. from a 1% aq. NH_3 soln., while warming, by the addition of sufficient K_2CrO_4 to give a concn. of 1% in the soln., the total vol. not exceeding 100 ml; the ppt. should be washed with 80% acetone or 50% ethanol until a negative reaction for CrO_4^{2-} is obtained.

C. D. KOPKIN

3656. **Anion-exchange studies of scandium(III) and vanadium(IV). Separation of scandium, titanium and vanadium.** R. I. Walter (Brookhaven Nat. Lab., Upton, L.I., New York). *J. Inorg. Nuclear Chem.*, 1958, **6** (1), 58–62.—The coarse residue from 100 to 200-mesh Dowex 1 (formate form) was used. Tervalent Sc and V^{IV} were strongly adsorbed from citrate and oxalate soln., V^{IV} was also strongly adsorbed from carbonate and fluoride soln., and Ti^{IV} was strongly adsorbed from oxalate soln. Scandium, V and Ti were adsorbed on a column from 0·1 *M* oxalic acid and then successively eluted, Sc with 0·1 *M* oxalic acid - 0·1 *M* HCl, V with 0·1 *M* oxalic acid - 0·4 *M* HCl, and Ti with 0·1 *M* HCl. G. J. HUNTER

3657. **Analysis of rare-earth elements. I. Micro-analysis of praseodymium, neodymium and samarium in lanthanum oxalate by spectrophotometry.** Giichi Muto and Masato Mamiya (Inst. of Ind. Sci., Tokyo Univ., Yayoicho, Chiba). *Japan Analyst*, 1958, **7** (1), 21–24.—Praseodymium, Nd and Sm (< 0·01%

each) in La oxalate are fractionally pptd. with 8-hydroxyquinoline (**I**) at pH 3·98, 4·38 and 4·58, respectively, whereas La is pptd. at pH 4·20. Most of the La can thus be separated to avoid interference with the spectrophotometric determination of these elements in HCl (*e.g.*, Kirk and Othmer, "Encyclopedia of Chemical Technology," 1953, Vol. 11, 504). The La oxalate (20 g) is ignited at 800° for 1 hr., dissolved in conc. HCl (50 ml), evaporated to dryness, diluted to 50 ml and mixed with **I** (30 g). The pH is carefully adjusted to 3·98 with aq. NH_3 soln. to precipitate Pr, which is photometrically determined in HCl soln. (5 ml) at 444·5 mμ. Lanthanum is removed at pH 4·20, and Nd (521·8 mμ) and Sm (402·0 mμ) are photometrically determined in the filtrate, after the removal of **I** by evaporation. K. SAITO

3658. **Use of a calculated calibration curve in the spectrographic analysis of the rare-earth elements by the method of additions.** A. I. Akimov. *Optika i Spektroskopiya*, 1957, **2** (6), 809–811; *Ref. Zhur., Khim.*, 1958, Abstr. No. 4293.—It is assumed that the index b in the formula $I = ac^b$ is known. The equation $\log I_1/I_2 = \log a + b \log (c + x)$, where x is the concn. of the element to be determined, and c is the known concn. added, is transcribed in the form $\log R_c/R_0 = b \log (I + c/x)$, where R_c is $\log I_1/I_D$ for the standard and R_0 is $\log I_1/I_2'$ for the base. For the analysis, a value of c is chosen such that the intensities of the lines in the spectra of the standard and the base are clearly distinguished. On the graph of co-ordinates $\log R_c/R_0$, $\log (I + c/x)$, choose a point the ordinate of which is equal to the value of $\log R_c/R_0$ found from the data of the photometry of the spectra of the standard and base. The abscissa of this point serves for the calculation of x, which is effected from a subsidiary curve on the co-ordinates $\log (I + c/x)$, x. For each standard a separate subsidiary curve is constructed. To determine x it is sufficient to have only one standard. The proposed method of calculation has been checked in the determination of Nd in samarium by analysis of their soln., with the use of an a.c. arc and a large spectrograph with a glass optical system. For the determination of x with triple photography of the spectra ≯ 0·6 mg of substance is necessary.
C. D. KOPKIN

3659. **Compensation method for the determination of actinium in small quantities.** V. I. Baranov. *Trudy Radiev. Inst., Akad. Nauk SSSR*, 1957, **8** (2), 31–36; *Ref. Zhur., Khim.*, 1958, Abstr. No. 874.—The applicability of the compensation method to the determination of Ac in soln. and in powder form (by its emanation) is studied. It is shown that the sensitivity (based on uranium) is respectively 5×10^{-5} and $8·1 \times 10^{-7}$ g of U, which at radioactive equilibrium corresponds to $1·7 \times 10^{-11}$ and $2·7 \times 10^{-13}$ g of Ra. In the analysis of natural substances it is recommended that the method of "added volume" be used in accordance with which a known vol. is included in the path of the air from the gas bubbler with the soln. to the ionisation chamber, which causes a lengthening of the time of passage of the emanation from the source to the ionisation chamber, and at the same time decreases the ionisation caused by Tn and An. Calculated formulae are given for the evaluation of I_{Tn} and I_{An}. The described method allows the determination of I_{An} corresponding to $7·5 \times 10^{-4}$ g of U ($2·5 \times 10^{-10}$ g of Ra) at a ratio of Th to U = 1·4.
C. D. KOPKIN

3660. **Gas-chromatographic determination of very small amounts of carbon disulphide in carbon dioxide and inert gases after preliminary enrichment.** J. Sverak and P. L. Reiser (Österreich. Stickstoffwerke A.-G., Linz, Austria). *Mikrochim. Acta*, 1958, (3), 426–433.—An apparatus is described for the determination of very small amounts (down to 1·5 mg per Nm^3) of CS_2 in carbon dioxide or inert gases. A preliminary enrichment stage is incorporated, in which the CS_2 is absorbed on "Blaugel" at 0° and subsequently removed by raising the temperature to 200°. The enriched gas is then passed through a chromatographic column containing dimethylsulpholane (20%) - sterchamol as absorber. The peaks are detected by a system which measures changes in thermal conductivity. The CS_2 present is determined by measuring the area of the peak, which is directly proportional to the amount of CS_2. The accuracy is ≃ ± 5% (relative) at concn. of 5 mg of CS_2 per Nm^3, but is reduced by the presence of SO_2. The enrichment stage might usefully be incorporated into other analytical procedures for the determination of CS_2.
S. M. MARSH

3661. **Mercurimetric titrations with redox indicators. II. The determination of thiocyanate, cyanide and mercuric ions.** J. Bognár (Tech. Univ. for the Heavy Industries, Miskolc, Hungary). *Magyar Kém. Foly.*, 1958, **64** (1), 37–40.—Halides, thiocyanate or cyanide are titrated in the presence of $Fe(CN)_6^{3-}$; when, at the end-point, an excess of Hg^{2+} is present, the oxidation potential of $Fe(CN)_6^{3-}$ increases considerably. Thus redox indicators with a high transition potential can be used; in the present detailed investigation, triarylmethane indicators were employed. Thiocyanates can be titrated even with a 0·02 *N* $Hg(NO_3)_2$ soln.; with a 0·01 *N* titrant, the accuracy is less. The determination can be carried out even in a 12 *N* H_2SO_4 soln. The effect of various ions is discussed. In order to determine Hg^{2+}, an excess of a thiocyanate soln. is added and the excess is back-titrated with $Hg(NO_3)_2$; this gives a sharper colour change than the direct titration. When CN^- are titrated with 0·1 *N* $Hg(NO_3)_2$, the accuracy is ± 0·2%; the use of a 0·02 *N* soln. also gives satisfactory results. A. G. PETO

3662. **Determination of silica. I. Spectrophotometric determination of silica as α-molybdosilicate.** L. H. Andersson (Inst. of Chem., Univ. of Uppsala, Sweden). *Acta Chem. Scand.*, 1958, **12** (3), 495–502.—Established methods for the spectrophotometric determination of Si by formation of molybdosilicate do not achieve completion of the change (slow at room temp.) from the unstable β- to the stable α-form, and the absorption is dependent on salt concn. and pH. In a new procedure, the subject silica soln. is mixed with molybdate soln. in a polyethylene bottle and heated on the steam bath for 3 hr. The bottle is then cooled and the contents are made up to volume at 20°, and the absorption is measured at 400 mμ. Since the max. absorption (at 350 mμ) corresponds to a high absorption of the blank, the absorption is measured at a longer wavelength (400 mμ is recommended) and the wavelength setting must be rigorously controlled. For high concn. of SiO_2, heating must be carried out for more than 3 hr. The absorption is nearly const. over the pH range 1 to 4·3. Concn. of MoO_4^{2-} in the reagent must be greater than the 12:1 ratio required for the heteropoly acid. Heating soln. 0·2 *M* with respect to Na_2MoO_4 may produce

ppt. and give a low extinction, but 0·05 to 0·1 *M* soln. are satisfactory. The absorption varies with temp. by ≃ 0·2 to 0·3% per °C. Beer's law is followed fairly closely. E. J. H. BIRCH

3663. **The reporting of chemical analyses of silicate rocks.** R. A. Chalmers and E. S. Page (Chem. Dept., Univ. Aberdeen, Scotland). *Geochim. et Cosmoch. Acta*, 1957, **11** (4), 247–251.—The statistical errors involved in a silicate analysis conducted by the classical method are enumerated and the presentation of the results of such an analysis is discussed. It is recommended that analyses be reported in such a way that the degree of precision of the analysis is included in the quoted results: in petrology such an estimate of the precision and accuracy of the results should be available so that the significance of the differences between analyses of different rocks may be properly assessed. R. A. HOWIE

3664. **The anion-exchange separation of tin and antimony.** J. Dawson and R. J. Magee (Dept. Chem., Queen's Univ., Belfast, N. Ireland). *Mikrochim. Acta*, 1958, (3), 325–329 (in English).—The sample soln. containing Sb^{V} and Sn^{IV} is adjusted to pH 4·8 and is passed through a 30-cm anion-exchange column (Amberlite IRA-400, 50 to 72 mesh, malonate form) at such a rate that the operation lasts 20 min. After a further 15 min. the Sb is eluted from the resin with ≃ 120 ml of 3% malonic acid soln. buffered to pH 4·8. The eluting soln. is then changed to 9 *N* H_2SO_4 and the Sn is removed at a higher rate of elution (1 ml per min. per sq. cm). The separation factor is high. W. J. BAKER

3665. **Semi-micro determination of tin and antimony in type metals.** A. A. Yavorovskiĭ and V. M. Shimanskiĭ. *Sb. Trud. Ukr. Nauch. Inst. Poligr. Prom.*, 1956, **4**, 127–134; *Ref. Zhur., Khim.*, 1958, Abstr. No. 964.—The standard method for the determination of Sn and Sb in type metals by the semi-micro method is modified. Dissolve the sample (0·1 g) by warming with conc. H_2SO_4 (3 ml), cool, add HCl (1 : 1) (10 ml), warm to a clear soln. and determine Sb by titration with 0·1 *N* $KBrO_3$ in the presence of 0·2% methyl orange soln. A blank is carried out simultaneously. Then to the soln. add 15% HCl (5 ml) and water (10 ml) and introduce a spiral of 30 cm of iron wire. Close the flask with a cork fitted with a Bunsen valve and warm, thereby dissolving the $PbSO_4$ ppt., reducing Sb^{5+} to metallic Sb and reducing Sn^{4+} to Sn^{2+}. Maintain the soln. near to boiling for 20 min., and filter into a flask containing CO_2 from marble and HCl. After cooling, determine the Sn by titration in an atmosphere of CO_2 with 0·2 *N* iodine to starch indicator. The time for the analysis is 1 hr. C. D. KOPKIN

3666. **Potentiometric determination of lead.** Ram Sahai Saxena (Gov. Coll., Kota, Rajasthan, India). *Z. anal. Chem.*, 1958, **160** (3), 194–197 (in English).—Lead can be titrated potentiometrically with $K_4Fe(CN)_6$ soln. containing 1% of $K_3Fe(CN)_6$. A sharp change in e.m.f. corresponds to the formation of $Pb_3Fe(CN)_6$. The error is about 1% in aq. soln. and less in the presence of 10 to 20% of ethanol. Electrolytes interfere. A platinum-foil electrode is used together with a S.C.E. connected through a saturated NH_4NO_3 bridge. G. BURGER

3667. **Alkalimetric titration of lead in frits, glazes, etc., after the addition of Complexone I (nitrilotriacetic acid).** W. Schmied and H. Steiner. *Glas-Email-Keramo-Tech.*, 1958, **9** (1), 6–7.—The formation of the complex of Pb with nitrilotriacetic acid (**I**) in a neutral nitrate soln. results in the liberation of a corresponding amount of acid which is titrated with alkali. Since all ions other than those of the alkali metals either give the same reaction or form a gelatinous ppt. (*e.g.*, Fe, Al, Cr, at pH 6) which interferes with the end-point, it is necessary to separate the Pb by pptn. as its thiourea nitrate. *Procedure*—The sample (1 to 1·5 g) is fused with 6 times its wt. of NaOH (a little KNO_3 is added to protect the platinum crucible). The melt is dissolved in HNO_3 soln. and, after separating the SiO_2, the filtrate is almost neutralised to methyl red, then made up to 250 ml, and an aliquot is titrated with 0·1 *N* NaOH. If interfering ions are present, the soln. (10 to 15 ml) from the fusion is mixed with an excess of solid thiourea and cooled to 0° for 30 min. before it is filtered in a Gooch crucible at 0°. The ppt. is washed twice with a soln. made by saturating *N* HNO_3 with thiourea, adding sufficient $Pb(NO_3)_2$ to form a slight ppt., cooling to 0° and filtering, and finally with the wash soln. made without the addition of $Pb(NO_3)_2$. The ppt. is dissolved in warm water and neutralised (methyl red) with NaOH soln. A few drops of 0·1 *M* **I** are added, and the soln. is titrated with 0·1 *N* NaOH. The addition of **I** and further titration is repeated until a red colour is no longer formed. (Excess of **I** during the titration is to be avoided.) The accuracy is within ± 1% of the true PbO content. J. A. SUGDEN

3668. **Phase analysis of complex titanium alloys.** N. I. Blok, A. I. Glazova, G. M. Kokhova and N. F. Lashko. *Zavod. Lab.*, 1958, **24** (2), 141–145.—Anodic dissolution is carried out in a bath containing 2 to 3 g of KSCN, 10 g of citric acid, 100 ml of glycerol and 1100 ml of methanol at a c.d. of 0·013 amp. per sq. cm and at − 7° to − 10°. The necessary membranes are prepared from a soln. of acetylcellulose in acetone. In the anodic ppt., Ti, Al, Cr and Mo are determined by normal methods. Phases rich in H have been isolated from certain alloys. G. S. SMITH

3669. **Determination of oxygen in titanium.** H. G. Lange von Stocmeier (Farbenfabriken Bayer, Leverkusen, W. Germany). *Arch. Eisenhüttenw.*, 1958, **29** (2), 95–100.—Methods for the determination of oxygen in titanium are reviewed. Application of a hot vacuum extraction apparatus designed for use with steel and the technique of Walter (*Anal. Chem.*, 1950, **22**, 297) was successful. E. G. CUMMINS

3670. **Annulus method for the separation and detection of titanium and chromium on a filter-paper.** V. M. Bhuchar and M. R. Verma (Nat. Phys. Lab., New Delhi, India). *Mikrochim. Acta*, 1958, (3), 342–346 (in English).—In the procedure described, five drops of the sample soln. are placed in the centre of a Whatman No. 1 filter-paper (Fe-free), which is then dried (i.r. lamp). Three drops of thioglycollic acid are added and the spot is heated for ≃ 10 min. (i.r. lamp). A deep-green coloration indicates Cr. A few drops of 5% aq. ammonium benzoate soln. and aq. NH_3 are then placed on the spot, which is again heated (steam-nozzle) to precipitate Ti as benzoate. The sol. Cr - thioglycollic acid complex can be eluted with

aq. NH_3 in the presence of thioglycollic acid to form an outer ring, whilst by dissolving the Ti benzoate in HCl, neutralising the excess of acid with aq. NH_3 and then adding a few drops of 0·1% chromotropic acid soln. a brown - red inner ring is formed, indicating Ti. The test is sensitive to 5 μg of Ti in the presence of 1000 μg of Cr, and 5 μg of Cr in the presence of 100 μg of Ti.

W. J. BAKER

3671. **Quantitative determination of zirconium with phytin.** I. P. Alimarin and L. Z. Kozel'. *Khimiya Redkikh Elementov*, 1957, (3), 114–118; *Ref. Zhur., Khim.*, 1958, Abstr. No. 7596.—Zirconium phytate, produced from phytin and Zr, is soluble only in oxalic acid and NaF, with formation of complex compounds. At 1000° to 1050°, it is converted into $Zr_2.2P_2O_6.P_2O_7$, which is suitable for weighing. A method for determining Zr with phytin is evolved, which is suitable for its determination in alloys and steel.

C. D. KOPKIN

3672. **Phosphate - 8-hydroxyquinoline method for the separation and volumetric determination of zirconium.** A. V. Vinogradov and V. S. Shpinel'. *Atomn. Energiya*, 1957, **3** (8), 130–134; *Ref. Zhur., Khim.*, 1958, Abstr. No. 4299.—The described method is a combination of the known phosphate method for the separation of Zr and a method for the determination of Zr as 8-hydroxyquinolinate. The conditions are studied for the separation of Zr from Ti and Th by phosphate pptn. and from Nb and Ta by 8-hydroxyquinoline pptn. To 50 or 60 ml of soln. (1 to 12 mg of Zr), 10% in H_2SO_4, add 3 to 5 ml of 30% H_2O_2 (when Ti is present) and a 10 to 100-fold excess (based on Zr) of a 10% soln. of $(NH_4)_2HPO_4$, heat to 80° or 90°, allow to stand, filter off the ppt., wash it with a cold 5% soln. of $(NH_4)_2HPO_4$ in 5% H_2SO_4 until aq. NH_3 does not give a cloudiness, and dissolve the residue in 10% oxalic acid soln. In the presence of Ti add to the wash liquor 1 or 2 ml of 30% H_2O_2 and wash the ppt. before its dissolution 3 or 4 times with the wash liquor without H_2O_2. To the oxalate soln. obtained, add 3 to 5 ml of a 3% 8-hydroxyquinoline soln. in 1% acetic acid and aq. NH_3 till pptn. is complete; in the presence of Nb and Ta add 60 or 70 ml of conc. aq. NH_3 immediately. Warm to between 70° and 80° till the ppt. has coagulated, cool, filter, wash the ppt. 3 or 4 times, first with hot (70° to 80°) and then with warm 5% NH_4Cl soln. till a clear colourless wash soln. is obtained, dissolve in hot 20% (v/v) HCl and wash the filter with the same acid. To the cooled soln. add 2 g of KBr and 3 or 4 drops of methyl red soln. and titrate with 0·05 or 0·01 *N* $KBrO_3$ till the colour changes from orange to a pure yellow. Add 2 or 3 ml in excess and 5 ml of a 20% soln. of KI and, after 3 to 5 min., titrate the liberated iodine with 0·05 or 0·01 *N* $Na_2S_2O_3$ in the presence of starch. The method determines 2 to 5 mg of Zr with an error of ± 4%. It ensures the separation of Zr from all accompanying elements, with the exception of Hf, and may be used in the analysis of any zirconium alloys, ores and intermediate products.

C. D. KOPKIN

3673. **Trace analysis. III. Trace analysis of zirconium and its compounds.** O. G. Koch (Neunkircher Eisenwerk A.-G., Neunkirchen, Saar). *Mikrochim. Acta*, 1958, (3), 347–352.—A method for separating and concentrating the trace elements in zirconium and its compounds is described. Zirconium is dissolved in H_2SO_4 - HF and the resulting mixture is taken up in HCl and diluted before analysis. Zirconium dioxide and barium zirconate may be dissolved by fusing with a mixture (3:2) of Na_2CO_3 and K_2CO_3 (1:1) and borax, and dissolving the melt in HCl. To the acid soln. is added 20 ml of 25% aq. ammonium tartrate soln. and the pH is adjusted to 3·0 with 6 *N* aq. NH_3. Extraction is then carried out with 2 ml of 5% aq. ammonium tetramethylenedithiocarbamate soln. and 15 ml of 0·01% dithizone in $CHCl_3$ (× 3 or 4) and the aq. residue is finally washed with 2 ml of carbamate solution and 15 ml of $CHCl_3$ until the $CHCl_3$ layer remains green. The pH is then adjusted to 5·0 with aq. NH_3 and the extraction is repeated. After a further addition of ammonium tartrate soln., the extraction is repeated at pH 7·0 and 9·0. The extracted material is ashed with addition of Be, which acts as a reference element, and the residue is dissolved in aqua regia and examined spectrographically. The following elements have been identified—Ag, As, Au, Bi, Cd, Co, Cr, Cu, Fe, Ga, Hg, In, Mn, Mo, Nb (incomplete extraction), Ni, Pb, Pd, Pt, Sb, Se, Sn, Te, Tl, U, V and Zn. The determination limit of the procedure is 0·00001% and accuracy is ± 15%.

S. M. MARSH

3674. **New gravimetric method for the micro-determination of thorium.** T. I. Pirtea and G. Mihail. *An. Univ. "C. I. Parhon," Ser. Ştiinţ. Natur.*, 1956, (12), 51–55; *Ref. Zhur., Khim.*, 1958, Abstr. No. 889.—The gravimetric determination of Th^{4+} by the use of the sodium salt of 2-mercaptobenzothiazole (**I**) (Spacu and Pirtea, *Bul. Ştiinţ. Acad. R.P. Române, Ser. Mat., Fiz., Chim.*, 1950, **2**, 8) is used for the micro-determination of Th. To 0·2 to 5 ml of the soln., containing ≃ 0·5 mg of Th^{4+} per ml, add 5 to 20 drops of a 10% soln. of **I**, stir and allow to stand for 5 min. Filter off the ppt., wash it 2 or 3 times with 0·5 ml of a 0·5% soln. of **I** and 3 or 4 times with 3 or 4 drops of water, and dry for 30 to 60 min. at 110° to 115°. The alkali metals and Mg do not interfere if they are in the form of salts of strong acids. The presence of free acid causes interference. The error of the determination is ± 2·2%.

C. D. KOPKIN

3675. **Complexometric micro-determination of thorium.** R. Přibil and E. Kőrös (Res. Inst. Czech. Acad. of Sci., Prague). *Magyar Kém. Foly.*, 1958, **64** (2), 55–57.—The pH of the soln. containing Th^{4+} is adjusted to between 1·5 and 3·5 by means of *N* HNO_3 or *N* aq. NH_3. (The lower the pH, the fewer ions interfere.) Add 0·1% xylenol orange soln. (*cf.* Körbl and Přibil, *Anal. Abstr.*, 1957, **4**, 2072) (3 to 6 drops) and titrate to yellow with a 0·01 *M* or 0·001 *M* soln. of EDTA (disodium salt). By using a 0·001 *M* soln., even a few 100 μg of Th^{4+} can be determined with good accuracy. Halides (except F^-), SO_4^{2-}, acetate, NH_4^+, alkali and alkaline-earth metals do not interfere; the interference caused by Fe^{3+}, Co, Ni and Cd cannot be eliminated, except by extracting the Th. At pH 2, the max. permissible amounts (in mg) of some other ions are—Zn^{2+}, 5; Al^{3+}, 10; La^{3+}, 10; Mn^{2+}, 100. At pH 3, 30 mg of UO_2^{2+} can be present; Pb^{2+} (30 mg) can be masked with KI, and Cu^{2+} (200 mg) with KSCN and ascorbic acid.

A. G. PETO

3676. **Determination of rare-earth impurities in thorium by spectrographic methods.** L. W. Wray (Atomic Energy Can. Ltd., Chalk River, Canada). *Atomic Energy Can. Ltd., Chalk River Project*, Rep. AECL-546, 1957, 14 pp.—The following

procedure is used to free rare-earth impurities from a thorium matrix before spectrographic examination. The sample is prepared so that 2 g of Th is contained in 20 ml of 6 *M* HNO_3 by either direct dissolution in the acid or by first refluxing for 1 hr. with 10 ml of conc. HNO_3 and 2 ml of 0·025 *M* NH_4F and then heating to dryness. When dissolution is difficult, HF is used, and the excess of fluoride is removed by fuming to dryness with 10 ml of concn. $HClO_4$. Most of the Th is removed by 2 extractions with 20 ml of 50% (v/v) tributyl phosphate in CCl_4. The aq. layer is washed with 20 ml of CCl_4, then 20 ml of benzene, and evaporated almost to dryness. The residue is dissolved in 40 ml of water and the pH is adjusted to 2·5 to 3·0 either by directly adding *M* HNO_3 or by heating to dryness, re-dissolving the residue in 40 ml of water, and adding the acid. The content of Th is further reduced by extraction with 40 ml of 0·5 *M* 2-thenoyltrifluoroacetone in benzene, followed by washing with 40 ml of benzene. The procedure results in an average recovery of rare earths of about 85%, as shown spectrographically. Fluoride adversely affects the removal of Th and the accuracy of the results. CHEM. ABSTR.

3677. **Assay of isotopic nitrogen by mass spectrometer.** A. P. Sims and E. C. Cocking (Dept. of Botany, Univ. of Bristol, England). *Nature*, 1958, **181**, 474.—Two methods are commonly used for correcting for the presence of air in nitrogen samples—(*i*) measurement of the oxygen peak at mass number 32, and use of a predetermined N:O ratio for air, (*ii*) measurement of the N:A ratio. With N produced in biological experiments, these methods are improved by purifying the nitrogen sample before passing it to the mass spectrometer. Thus, errors due to O, formed by the decomposition of NaBrO, are prevented by adding KI to the NaBrO reagent, and other impurities such as CO_2 and H_2O are frozen out by liquid air.

G. S. ROBERTS

3678. **Determination of nitrogen in metals by the combustion method. I. Nitrogen and carbon in steel and titanium.** Masahiro Tsuchiya (Inst. of Techno-anal. Chem., Fac. of Engng, Tokyo Univ., Hongo). *Japan Analyst*, 1958, **7** (1), 12–17.—The combustion method followed by mass spectrometry was applied to the determination of N in iron, steel and sponge titanium. The combustion of the nitrides of Fe, Al and Ti is complete below 700°, 900° and 1100°, respectively, whilst chromium nitride is only partly converted into N with PbO as flux (for the apparatus, see Klinger, *Arch. Eisenhüttenw.*, 1931, **5**, 29). The combustion of C in these samples in a current of O is complete below 1100° when samples of < 10 mesh are used.

II. Nitrogen and carbon in ferro-alloys. Masahiro Tsuchiya. *Ibid.*, 1958, **7** (1), 17–21.—Nitrogen in ferrochromium (0·3 g) is satisfactorily liberated within 30 min. by heating the sample (< 100 mesh) in a current of O (50 ml per min.) at ⩾ 1200° together with a mixture (5 times the wt. of the sample) of Pb_3O_4 and B_2O_3 (3 + 1, by wt.) as flux. Nitrogen in ferrosilicon is readily converted into N gas in the presence of PbO at ⩾ 1200°.

K. SAITO

3679. **Vanadametry. Determination of hydroxylamine. Use of copper sulphate as catalyst.** H. S. Gowda, K. Bhaskara Rao and G. Gopala Rao (Andhra Univ., Waltair, India). *Z. anal. Chem.*, 1958, **160** (2), 117–120 (in English).—The oxidation of hydroxylamine to N_2O by Na_3VO_4 is rapid and quantitative in 5 to 6 *N* HCl in the presence of $CuSO_4$. Excess of Na_3VO_4 can be titrated with $(NH_4)_2SO_4.FeSO_4$ soln. to determine hydroxylamine.

G. BURGER

3680. **The spectrochemical determination of phosphorus in steel by the use of the phosphorus line at 3175 A.** S. Eckhard (Max Planck Inst., Düsseldorf, W. Germany). *Arch. Eisenhüttenw.*, 1958, **29** (2), 89–94.—Emission in the region 3174·96 to 3175·447 A and the spectrum of phosphorus between 2484·24 and 4130·92 A are discussed.

E. G. CUMMINS

3681. **Rapid determination of phosphate in ore and calcium superphosphate.** Shizo Hirano and Hiroshi Kawaguchi (Inst. Techno-anal. Chem., Fac. of Engng, Tokyo Univ., Hongo). *Japan Analyst*, 1958, **7** (2), 107–108.—The determination of orthophosphate with $AgNO_3$ followed by alkalimetric titration of the resulting HNO_3 (Gerber and Miles, *Ind. Eng. Chem., Anal. Ed.*, 1941, **13**, 406; van Wazer *et al.*, *Anal. Abstr.*, 1955, **2**, 902) was applied to phosphate ore and calcium superphosphate, with the aid of EDTA as masking agent for Ca, Al and Fe. Interference results from Bi, Hg, Sb, Ag, Pb (these are rarely found in the sample), As and Cr. *Procedure*—Decompose the sample (0·4 g) with $HClO_4$ (60%, 5 ml), neutralise with NaOH (indicator, 0·1% bromocresol green and 0·2% methyl red, 1 + 7, by vol.), make slightly acid with *N* $HClO_4$ and add 4% EDTA (disodium salt) until the soln. is clear. Adjust the pH to 4·5 with 0·1 *N* NaOH, add $AgNO_3$ soln. (10%, 10 ml) and titrate with 0·1 *N* NaOH to bromothymol blue (0·1%, 10 drops).

K. SAITO

3682. **Infra-red absorption spectra of various phosphorus compounds. II. Alkali pyrophosphates.** A. Mutschin and K. Maennchen (Chem. Fabrik Zschimmer & Schwarz, Oberlahnstein/Rhein). *Z. anal. Chem.*, 1958, **160** (2), 81–98.—The spectra of the alkali pyrophosphates have been studied in the regions 1 to 15 μ (NaCl region), 15 to 25 μ (KBr region), and 25 to 40 μ (CsBr region) by the potassium bromide pellet method with 3 to 6 mg of pyrophosphate in 1000 mg of KBr. Neutral and acid salts can be distinguished by the shift of the OH bands. The effect of water of crystallisation is discussed. The symmetrical and asymmetrical POP bands are specific for condensed phosphates. Anomalies in the potassium bromide pellet method are discussed.

G. BURGER

3683. **Determination of arsenic in microgram quantities in coal and coke.** A. Crawford, J. G. Palmer and J. H. Wood (Central Lab., Nat. Coal Board, Manchester, England). *Mikrochim. Acta*, 1958, (2), 277–294 (in English).—The method is based on an instrumental comparison of the light-absorptive properties of the blue molybdoarsenate complex formed with similar soln. containing known amounts of As. Organic matter is wet-oxidised and As is brought into soln. by treatment with HNO_3 and H_2SO_4. The As is reduced to the tervalent state and evolved as arsine by the action of Zn and H_2SO_4. It is absorbed and oxidised to arsenic acid by dil. iodine soln., which, by treatment with ammonium molybdate soln. and reduction with hydrazine sulphate, is converted into the blue molybdoarsenate complex. The optical density of the soln. measured at 835 mμ is proportional to the As content of the sample. Apparatus for the wet oxidation and for the evolution and absorption

of arsine is described and illustrated. Satisfactory reproducibility is shown for the method when applied to 8 coals, 18 anthracites and 3 cokes in the range 0·8 to 17·1 μg of As.

D. F. PHILLIPS

3684. **Influence of copper and lead on the bromatometric determination of antimony.** T. Chlebovský (Vysoká Škola Báňská, Ostrava, Czechoslovakia). *Hutn. Listy*, 1958, **13** (2), 148–150.—The influence of Cu can be eliminated only by a preliminary separation before Sb is titrated. In the presence of large amounts of Pb, an adsorption of Sb^{3+} on $PbSO_4$ was observed; a titration of Sb^{3+} in the ppt. of $PbSO_4$ (after dissolution in HCl) must therefore also be carried out. J. ZÝKA

3685. **Polarographic determination of antimony in washed concentrates [of copper - antimony].** M. Spálenka (Res. Inst. Metals, Panenské Břežany, Czechoslovakia). *Hutn. Listy*, 1958, **13** (2), 150–151.—*Procedure*—To the sample (2 to 5 ml containing ⩾ 8 to 10 mg of Sb) in a 50-ml flask add H_2O_2 soln. (30%) dropwise till the reaction is finished. Then add conc. HCl (2 to 5 ml) saturated with Br. Remove the excess of Br with heating, cool, and add aq. NH_3 (1:1) (5 ml). Heat, cool, add hydrazine sulphate (satd. soln.) (1 ml), gelatin soln. (0·5%) (1 ml) and conc. HCl (25 ml) and dilute with water. Register the anodic - cathodic wave from 0 to 0·5 V. The determination takes ⩾ 35 min. The accuracy is within ± 3%. J. ZÝKA

3686. **Distillation of antimony halides and the colorimetric determination of antimony with methyl violet.** Yu. V. Morachevskiĭ and D. G. Barbanel'. *Uch. Zap. LGU*, 1957, (211), 76–82; *Ref. Zhur., Khim.*, 1958, Abstr. No. 925.—Satisfactory results were obtained in the analysis of copper alloys by this method by the use of the photometric determination of Sb with methyl violet, the error being ≃ 0·0002%. C. D. KOPKIN

3687. **The determination of antimony, tin and lead.** J. Dawson and R. J. Magee (Dept. of Chem., Queen's Univ., Belfast, N. Ireland). *Mikrochim. Acta*, 1958, (3), 330–336 (in English).—In the rapid method described, which is applicable to white-metal and tin alloys, the soln. containing Pb, Sn^{IV} and Sb^{V} is passed through a column of Amberlite IRA-400 (*cf. Anal. Abstr.*, 1958, **5**, 3664). After 15 min., Pb and Sb are removed from the column by elution with 120 ml of 3% malonic acid soln. (pH 4·8) at a rate of 0·5 ml per min., followed by elution with ≃ 100 ml of 9 *N* H_2SO_4 at 1 ml per min. to remove Sn. The three metals are then determined spectrophotometrically in separate aliquots of the eluates, Sb as iodoantimonate, Sn as iodostannate and Pb as the dithizone complex (Sb does not interfere). Large amounts of Cu should be removed initially, but concn. of ⩾ ≃ 5% can be tolerated provided that hydroxylamine hydrochloride is added before the addition of KCN in the determination of Pb. The accuracy is satisfactory.

W. J. BAKER

3688. **Determination of antimony in high-purity lead.** Yu. Yu. Lur'e and T. V. Zaglodina (State Inst. of Non-ferrous Metals). *Zavod. Lab.*, 1958, **24** (2), 133–134.—To determine 0·5 to 1 μg of Sb in a sample of 1 g of lead, the sample is dissolved in 10 ml of dil. HNO_3 (1:3) and the soln., after addition of 5 ml of dil. HCl (3:1), is evaporated to dryness. Following a second evaporation with 5 ml of dil. HCl the residue is dissolved in 10 ml of dil. HCl (3:1) and the soln. is treated with one or two drops of a 10% soln. of $SnCl_2$ in conc. HCl and cooled. If the colour is yellow a further drop is added. Then 1 ml of 10% $NaNO_2$ soln. is added followed, after 5 min., by 10 ml of water, 1 ml of a saturated soln. of urea, and a further amount of water to give 75 ml. The soln. in a separating funnel is treated with 13 to 15 drops of a 0·2% aq. soln. of crystal violet and 7 ml of toluene and shaken for 1 min. The extinction of the blue extract is measured with a 530-mμ filter.

G. S. SMITH

3689. **Determination of metallic impurities in ultra-pure antimony by means of the dithizone method.** E. Häberli (Siegfried A.-G., Zofingen, Switzerland). *Z. anal. Chem.*, 1958, **160** (1), 15–20.—The total metallic impurity present in amounts of the order of 0·1 μg in 1 g of antimony of semiconductor purity can be determined with sufficient precision (± 20 to 30%) by extractive titration with dithizone. The sample is treated with Br and dissolved in Na K tartrate soln. The pH is adjusted to 7·5 and the soln. is shaken with successive small quantities of dithizone soln. until no further colour change is observed in the extract. The vol. of dithizone soln. used is measured. A soln. containing Zn (0·3 μg) is added to the soln. of Sb and the extraction is repeated as a measure of the sensitivity. The procedure must be carried out in a clean atmosphere, all reagents must be specially purified and tested with dithizone, and apparatus must be washed with hot ammoniacal EDTA soln. A quartz vessel must be used when dissolving the sample. G. BURGER

3690. **Colorimetric determination of small quantities of antimony in copper-based alloys.** Yu. V. Morachevskiĭ and D. G. Barbanel'. *Uch. Zap. LGU*, 1957, (211), 62–75; *Ref. Zhur., Khim.*, 1958, Abstr. No. 924.—It is established that for complete separation of Sb by hydrated MnO_2, irrespective of the quantity taken, ⩽ 0·035 g of the collector is required. This quantity is about a quarter of that generally accepted. In separating small quantities of Sb (25 to 150 μg) with hydrated MnO_2 from soln. with a large content of Cu, low results are obtained, because of the weakening influence on the colour of the methyl violet complex with $SbCl_6^-$ of Cu which is co-pptd. with the carrier. The analysis of copper alloys by co-pptg. Sb with metastannic acid with subsequent photometric determination of Sb with methyl violet is recommended; the error of the method is ≃ 0·0002%. C. D. KOPKIN

3691. **Amperometric determination of bismuth with pyrogallol.** T. K. Musina and O. A. Songina. *Izv. Akad. Nauk KazSSR, Ser. Khim.*, 1957, (1), 36–44; *Ref. Zhur., Khim.*, 1958, Abstr. No. 4319.—Neutralise the acid soln. of Bi with aq. NH_3 to cresol red and titrate with a soln. of pyrogallol (**I**) at an applied voltage of + 1·0 V. Under these conditions Bi does not enter into the electrode reaction and forms Bi pyrogallate with **I**. At the end-point there is a sharp increase of current because of anodic oxidation of excess of **I**. The amount of **I** added up to the point of increase in current corresponds to the stoicheiometric quantity indicated by the formula $BiC_6H_6O_3$. The error of the determination is ± 4%. Interference is caused by Ag, Cu, Cd and Zn, but not by Pb.

C. D. KOPKIN

3692. **Metal - dye compounds as analytical reagents: determination of bismuth.** M. P. Grotheer (Kansas State Coll., U.S.A.). *Dissert. Abstr.*, 1957, **17** (11), 2412–2413.—A modified continuous-variation method was developed and used for the determination of the ratios of composition of various insoluble metal - dye compounds. Dilute soln. of thorium ions at pH 4·1 to 4·4 and of bismuth ions at pH 2·7 form compounds with sulphonic acid dyes, the positions of reaction being the same in number as the SO_3H groups. In an investigation of ion-exchange characteristics, soln. of various ions were allowed to react with the metal - dye compounds; it was found that Th - Amaranth has much the same ion-exchange selectivity on cellulose as on a homogeneously pptd. $Th(OH)_4$ medium; Bi compounds behave differently. I.r. spectra of the Amaranth compounds of Bi, Th and Zr showed that Bi and Th are bonded only with the sulphonate groups, but that Zr may form a chelate structure with the azo linkage and the adjacent hydroxyl group. Bismuth and Th form compounds with a ratio of 3 metal ions to 1 dye anion, and for Zr the ratio is 4 metal ions to 1 dye anion. At pH values below which complete hydrolysis occurs, Bi, Th and Zr form compounds of definite composition with the sulphonic acid dyes studied. To determine Bi in alloys, Bi is pptd. as Bi - Amaranth from a 1% boric acid soln. containing HNO_3 at pH 2·3 to 3·0. The compound is filtered off and washed with 1% boric acid at pH 3·0 to 3·5. The Amaranth is liberated by adding a known volume of Na_2HPO_4 through the compound on the filter. The absorbance of the released Amaranth soln. is measured and the concn. of Bi determined from a calibration curve. S. C. I. Abstr.

3693. **Detection of vanadium in qualitative analysis with ferric thiocyanate.** V. L. Zolotavin and A. F. Ogarkova. *Trudy Ural'sk. Politekh. Inst.*, 1957, (69), 148–150; *Ref. Zhur., Khim.*, 1958, Abstr. No. 21,175.—A new method is described for the detection of V based on the decolorisation of ferric thiocyanate as a result of reduction of Fe^{3+} in the presence of V^{II} and V^{III}. C. D. Kopkin

3694. **Complexones in chemical analysis. LIII. Photometric determination of niobium and tantalum with catechol and EDTA.** V. Patrovský (Centr. Geolog. Inst., Prague). *Chem. Listy*, 1958, **52** (2), 255–261.—Niobium and tantalum in the presence of EDTA and tartrate or oxalate form coloured complexes at about pH 2·5. The complex of Ta is yellow and has a max. in the u.v. range; that of Nb is red (470 mμ). The photometric determination of Ta and Nb in a mixture of their oxides in the presence of some impurities has been developed. *Procedure*—Fuse the sample (Nb:Ta ≯ 1:5) (0·2 g) with $KHSO_4$ (3 g) moistened with conc. H_2SO_4. When Nb:Ta is > 1:5, a preliminary separation with tannin must be carried out. *Niobium*—To the cooled melt add tartaric acid (1 g) and water (5 ml) and heat until dissolved. Dilute to a vol. of 25 to 50 ml with water. Measure a suitable amount (≡ ≯ 0·7 mg of Nb_2O_5) into a small vessel, add 0·1 *M* EDTA (disodium salt) (5 ml), tartaric acid (5%) (0·5 ml) and catechol (10%) (5 ml), adjust the pH to about 2·5 by adding formic or acetic acid, and boil for 30 sec. Cool, transfer to a 25-ml flask, make up to vol. with H_2O, mix, and measure the extinction at 470 mμ (blue filter). *Tantalum*—To the cooled melt add ammonium oxalate (1 g) and H_2O (5 ml) and heat until dissolved. Cool, and dilute to a vol. of 25 to 50 ml with a satd. soln. of ammonium oxalate. Measure a suitable amount (≡ ≯ 3 mg of Ta_2O_5) into a 25-ml flask, add 0·1 *M* EDTA (disodium salt) (5 ml), catechol (10%) (5 ml), ammonium oxalate (0·5 g) and acetic acid to a pH of about 2·5, make up to vol. with a satd. soln. of ammonium oxalate, and mix. Measure the extinction at 490 mμ (violet filter). The presence of Ti, W and Fe in 3 to 5-fold excess has no influence on the accuracy of the determination. The constitution of the complexes is discussed. J. Zýka

3695. **Extraction of the 8-hydroxyquinolinates of niobium, tantalum and tungsten.** I. P. Alimarin and I. M. Gibalo. *Vestn. Moskov. Univ., Ser. Matem., Mekhan., Astron., Fiz., Khim.*, 1956, (2), 185–188; *Ref. Zhur., Khim.*, 1958, Abstr. No. 7540.—The 8-hydroxyquinolinate of Nb is easily extracted with $CHCl_3$, dichloroethane, ethyl acetate, *cyclo*hexanone, butyraldehyde, *n*-butanol and *iso*amyl alcohol at pH 6 to 9 from soln. containing 2·5% of ammonium citrate or tartrate. In preparing the melt, take an 8 to 10-fold excess of $K_2S_2O_7$. Under these conditions the tantalum complex is not extracted by *iso*amyl alcohol, and the tungsten complex is not extracted by any of the solvents indicated. With the ratio of Nb to Ta of 1:1000, *iso*amyl alcohol extracts 89·1% of the Nb and none of the Ta. A quant. separation of Nb from W can be made with $CHCl_3$. For controlling the extraction, use was made of the isotopes ^{95}Nb, ^{182}Ta and ^{185}W. The extraction of Nb from oxalate soln. takes place to only a small extent. C. D. Kopkin

3696. **The separation of niobium, tantalum and titanium from ferrotantaloniobium.** E. Lassner and R. Scharf (Metallwerk Plansee G.m.b.H., Reutte/Tirol, Austria). *Z. anal. Chem.*, 1958, **160** (1), 21–22.—The sample (0·1 g) is dissolved in 5 ml of HNO_3 with the minimum amount of HF. The soln. is diluted with 20 ml of H_2O, and glycerol (15 ml) and 4% aq. H_3BO_3 soln. (50 ml) are added. A mixture of 25% aq. NH_3 (150 ml) and 2 g of solid EDTA is added and the soln. is boiled for 2 to 3 min. and filtered. The ppt. is ignited at 1000°. The results agree with those obtained by pptg. the Fe with $(NH_4)_2S$ in the presence of tartaric acid and then pptg. the Nb, Ta and Ti with cupferron and igniting to the oxides. G. Burger

3697. **Purification of protactinium by chromatography and electrophoresis on paper.** M. Lederer and J. Vernon. *Compt. Rend.*, 1957, **244** (19), 2388–2390.—The impurities that usually accompany Pa are Ta, Nb, Zr and Ti. Separation of Pa from Zr and Ti can be achieved by electrophoresis on paper with an aq. soln. of HF (5%) and HCl (5%), and from Ta and Nb by paper chromatography with a mixture of butanol, 12 *N* HCl, 20 *N* HF and water (50:25:1:24). G. Burger

3698. **Continuous coulometric determination of oxygen in a gas sample.** Isamu Sakamaki and Shinzo Yuki (Kurosaki Factory, Mitsubishi Chem. Ind., Yawata, Fukuoka-ken). *Japan Analyst*, 1958, **7** (1), 33–37.—The sample of gas containing < 5% of O is introduced into $CrCl_2$ soln. (2·5% in 10% KCl) (200 ml) (rate of flow 20 to 80 ml per min.) which is being electrolysed with a mercury cathode. The electrolytic current is so adjusted that a very small and known concn. of Cr^{2+} is maintained in the aq. soln., by the use of a servo-mechanism. The intensity of the electrolytic current indicates

the amount of O introduced into the aq. phase per unit of time; the O in the sample is thus determined at a given rate of flow. K. SAITO

3699. **Use of low-energy betatron radiation for the determination of oxygen in semi-conductors and metallic materials (particularly titanium oxycarbides).** A. Kh. Breger, B. F. Ormont, V. S. Kutsev, B. I. Viting and B. A. Chapÿzhnikov. *Zhur. Neorg. Khim.*, 1957, **2** (3), 696–699; *Ref. Zhur., Khim.*, 1958, Abstr. No. 928.—The reaction $^{16}O(\gamma, n)^{15}O$ is employed. For the irradiation, betatron radiation of max. energy 20 MeV with a dosage of 7 roentgen per min. at a distance of 1 metre from the target is used. The samples for study were prepared by compressing a powder containing 60 to 80% of Ti, 1 to 20% of O, 7 to 20% of C and 2% of liquid paraffin as a binder. The irradiation was carried out for 6 min. at a distance of $\simeq$ 30 cm from the target. Measurement of the activity was made with an end-window tube 30 sec. after the conclusion of the irradiation. A preliminary calibration curve for the concn. region 1 to 20% of O was obtained. C. D. KOPKIN

3700. **Determination of sulphur with the use of ion exchange.** O. Kšír, J. Jankovský and K. Follpracht (Inst. Res. Ores, Prague). *Rudy*, 1958, **6** (2, Add. 1), 1–3.—*Procedure*—Dissolve the sample (1 g) in a mixture of HNO_3 and HCl (3 + 1) (40 ml) (add bromine when necessary), transfer to a dish and evaporate to dryness on a water bath. Remove the excess of HNO_3, dissolve the residue in conc. HCl (2 ml) and hot water, and filter. Pour 50 ml of the filtrate through a column of Catex FN (3 to 4 ml per min.) and wash the column with water (100 ml); determine the separated sulphate gravimetrically, as $BaSO_4$, or titrimetrically. *Titrimetric procedure*—To the separated sulphate add alizarin red S (0·1%) (1·2 ml), neutralise dropwise with NaOH soln. (10%) till violet. Add acetic acid (1 : 3) (12 ml) and the same vol. of methanol. Titrate with 0·05 *M* $BaCl_2$, with stirring, till pink. J. ZÝKA

3701. **Volumetric determination of sulphate ions.** L. Légrádi and S. Füsti Molnár (Veszprém County Paint Works, Fűzfőgyártelep, Hungary). *Magyar Kém. Foly.*, 1958, **64** (1), 29–30.—To the soln. to be determined, $BaCl_2$ is added, and its excess is back-titrated with EDTA (disodium salt). *Procedure*—To the acid or neutral soln. containing SO_4^{2-} (0·03 to 0·04 g) add 0·02 *M* $BaCl_2$ (25 ml), the buffer [NH_4Cl (8·25 g) and conc. aq. NH_3 (113 ml) in 1 litre of aq. soln.] (2 ml), Eriochrome black T indicator [0·5 g in water (15 ml) and *N* Na_2CO_3 (0·5 ml) diluted to 100 ml with *iso*propyl alcohol] (2 drops) and freshly prepared Na rhodizonate indicator (5 drops of a 0·4% soln.). Titrate the soln. with 0·01 *M* EDTA (disodium salt) till the colour changes from red to a medium green. If 2 and 6 drops of the indicators are added, the colour is paler green; if 3 and 5 drops are used, it is a darker green. Titrate the 0·02 *M* $BaCl_2$ soln. (25 ml) separately with the EDTA soln. and from the difference of the two titrations the result can be calculated. When determining $CuSO_4$, $CdSO_4$ and $NiSO_4$, add KCN (0·5 g) after the $BaCl_2$. Ions of the following metals interfere—Pb, Hg^1, Bi, As, Sb, Sn, Cu, Fe, Mn and alkaline earths; also all anions that give a ppt. with Ba. A. G. PETO

3702. **Determination of sulphate ions with EDTA. I. Volumetric determination of sulphate ions with barium - EDTA complex.** Nobuhiko Iritani and Tomoo Tanaka (Shizuoka College of Pharm., Ojika, Shizuoka). *Japan Analyst*, 1958, **7** (1), 42–46.—The indication of the end-point of the EDTA titration with phthalein complexone (**I**) is improved by the use of the Ba - EDTA complex. The sample soln. containing 5 to 50 mg of SO_4^{2-} is adjusted to between pH 9 and 10, warmed with a slight excess of 0·02 to 0·1 *N* Ba - EDTA on a water bath and filtered. The filtrate is neutralised with dil. NaOH soln., then mixed with aq. NH_3 soln. (25%, 7 to 10 ml) and **I** (0·18% in ethanol containing 0·02% naphthol green) (a few drops) and titrated with 0·02 *N* $BaCl_2$. No interference results from CO_2. K. SAITO

3703. **Methods of determining sulphate impurities in soluble phosphates.** G. V. Zavarov (M. I. Kalinin Chernorechensk Chem. Works). *Zavod. Lab.*, 1958, **24** (2), 153–156.—In determining SO_4^{2-} in the presence of PO_4^{3-}, the pptn. of $BaSO_4$ can be carried out at high acidity (0·5 *N*) to reduce co-pptn., but good results, if obtained, are probably due to compensation of errors. In any case, filtration of the $BaSO_4$ must be postponed to the following day. Of various methods for prior removal of PO_4^{3-} with Mg^{2+}, the best are pptn. from a hot soln. by stirring with a mixture of $MgCl_2$ and NH_4Cl soln., and dropwise addition of $MgCl_2$ soln. to the well-stirred cold soln. Details of the various methods and results are given. G. S. SMITH

3704. **Separation of sulphate and tellurate ions by ion exchange.** Kenjiro Kimura, Nagao Ikeda, Mariko Inarida and the late Haruko Kawanishi (Chem. Dept., Fac. of Sci., Tokyo Univ., Hongo). *Japan Analyst*, 1958, **7** (2), 73–76.—Whilst SO_4^{2-} (< 10 mg) are adsorbed on Amberlite IRA-400 resin (Cl) ($\simeq$ 100 mesh) from a neutral or 0·1 *N* HCl soln., TeO_4^{2-} (< 10 mg) pass through. The SO_4^{2-} are eluted with NaCl soln. (10%, 30 ml). This method is applicable to the radiochemical analysis of fission products, especially for the detection of neutron-induced ^{35}S. The eluate from a sample of fission products in 0·2 *N* HCl, after passage through a column of Amberlite IR-120 (H), is boiled with $\simeq$ 4 *N* HNO_3 to remove iodine, and $HClO_4$ to remove Ru, and treated with $BaCl_2$. The resulting $BaSO_4$ is fused with Na_2CO_3, the melt is dissolved in water, then filtered, and the filtrate is passed through the resin column. K. SAITO

3705. **Spectrographic determination of selenium.** W. Rockenbauer and M. Brandenstein (Chem. Lab., Bundesversuchs- und Forschungsanstalt Arsenal, Vienna). *Z. anal. Chem.*, 1958, **160** (2), 104–108.—The concn. of Se in sulphide ores (0·0001% to 0·1% of Se) can be determined within $\pm$ 15% on 1 g of sample by a comparison of the Se lines at 2039·85 A, 2062·79 A and 2074·79 A with the S line at 2170·48 A produced in a d.c. arc spectrogram at 10 amp. and 110 V. The sample is finely ground and used without further preparation. G. BURGER

3706. **Trace analysis. IV. Trace analysis of selenium of high purity.** O. G. Koch (Neunkircher Eisenwerk A.-G., Neunkirchen, Saar). *Mikrochim. Acta*, 1958, (3), 402–405.—It has been shown that the reagents previously used for the extraction of trace elements from metals, *viz*, ammonium tetramethylenedithiocarbamate and dithizone in $CHCl_3$, react with selenium, which is therefore also extracted to some degree. A mixture of soln. of 8-hydroxyquinoline (0·1%) and dithizone (0·01%, in $CHCl_3$)

is a suitable medium for extracting trace elements from selenium. The recommended procedure involves stepwise extraction at pH 3·0, 5·0, 7·0 and 9·0, as previously described for the separation of trace elements from zirconium (*Anal. Abstr.*, 1958, **5**, 3673). The extracted material is then examined spectrographically. The following elements have been identified—Sc, Y, La, Ti, Zr, Hf, Th, V, Mo, U, Mn, Fe, Co, Ni, Pd, Pt, Cu, Ag, Au, Zn, Cd, Hg, Al, Ga, In, Tl, Sn, Pb, Sb and Bi. The accuracy of the method is ± 10% and the lower detection limit is 0·00001%. If a larger sample is used and suitable precautions are taken, this limit can be reduced to about 0·000001% to 0·0000005%.

S. M. MARSH

3707. **Use of extraction for increasing the sensitivity of the colorimetric determination of chromium with 1-naphthylamine.** V. N. Polyanskiĭ. *Sb. Trud. Mosk. Vech. Metallurg. Inst.*, 1957, (2), 253–254; *Ref. Zhur., Khim.*, 1958, Abstr. No. 11,016.—The sensitivity of the photometric method for determining Cr with 1-naphthylamine may be increased by extraction of the coloured complex with *iso*amyl alcohol. To the sample soln. in a separating funnel add a soln. of the reagent (0·5 g of 1-naphthylamine dissolved in a small quantity of acetone, to which soln. is added 50 g of tartaric acid dissolved in 100 ml of water). Add 5 or 10 ml of *iso*amyl alcohol, shake for 30 sec., and measure the extinction of the organic layer. The sensitivity of the determination is 10^{-4} g of Cr per litre, as compared with 10^{-3} g of Cr per litre without the use of extraction. C. D. KOPKIN

3708. **Extraction of chromium with trioctylphosphine oxide.** J. C. White and W. J. Ross (Oak Ridge Nat. Lab., Oak Ridge, Tenn.). *U.S. Atomic Energy Comm.*, Rep. ORNL-2326, 1957, 37 pp.—The extraction of Cr^{6+} from 1 to 7 *M* HCl or from 1 to 4 *M* H_2SO_4 with a 0·1 *M* soln. of tri-*n*-octylphosphine oxide (**I**) in *cyclo*hexane is quant., but Cr^{3+} are not extracted. The amount of Cr extracted quant. by 0·5 millimole of **I** is 12 and 20 mg from HCl and H_2SO_4, respectively. A phase vol. ratio, aqueous to organic, of 10 for HCl and 3 for H_2SO_4 is tolerable. The presence of an excess of anion retards the extraction. The species extracted from HCl and H_2SO_4 is $H_2Cr_2O_7.2\mathbf{I}$. The extraction from HNO_3, H_3PO_4 and $HClO_4$ is incomplete. CHEM. ABSTR.

3709. **Use of the amperometric method for the analysis of chromites and chromomagnesitic refractory materials.** E. G. Kondrakhina, L. G. Egorova and O. A. Songina. *Izv. Akad. Nauk KazSSR, Ser. Khim.*, 1957, (1), 45–50; *Ref. Zhur., Khim.*, 1958, Abstr. No. 7647.—To determine Cr and total Fe, fuse the sample with a mixture of Na_2O_2 and Na_2CO_3 and extract the melt with hot water. Filter off the ppt. of $Fe(OH)_3$, wash it with 1% Na_2CO_3 soln. and dissolve it in hot 2 *N* H_2SO_4. Reduce Fe^{3+} with metallic bismuth and titrate an aliquot of the soln. amperometrically with 0·1 *N* or 0·01 *N* $K_2Cr_2O_7$ at an e.m.f. of + 1·0 V *vs.* the mercury iodide electrode. Boil the filtrate for 20 min., neutralise with H_2SO_4, add excess of acid to a concn. of 0·1 *N*, and dilute to a known volume. Determine Cr in an aliquot by titration with 0·1 *N* or 0·05 *N* $(NH_4)_2SO_4.FeSO_4$. To determine FeO, place 0·4 g of V_2O_5 in a 100-ml beaker moistened with 1 to 1·5 ml of water, add 10 ml of a mixture of conc. H_3PO_4 and H_2SO_4 (1:2) and heat to dissolve; or use an aliquot of a soln. obtained by dissolving 4 g of V_2O_5 in 100 ml of the acid mixture and diluting to one litre. Add to the same beaker 0·1 to 0·25 g of chromite and heat to complete dissolution (5 to 15 min.). Dilute the soln. to 100 or 200 ml and titrate an aliquot amperometrically with a soln. of $(NH_4)_2SO_4.FeSO_4$ at an e.m.f. of + 1·0 V. Carry out a control experiment. C. D. KOPKIN

3710. **Simultaneous potentiometric determination of chromium and vanadium.** G. Piccardi (Inst. Anal. Chem., Univ. Florence). *Ann. Chim., Roma*, 1958, **48** (2), 188–192.—The Cr and V in the sample are oxidised to Cr^{6+} and V^{5+} and the mixture is titrated potentiometrically with 0·1 *N* $FeSO_4$. The mixture is then titrated potentiometrically with 0·01 *N* $KMnO_4$, the V being selectively re-oxidised to V^{5+}. Typical analyses of Fe - Cr - V mixtures in proportions corresponding to those in samples of steel are reported. The error varied from − 0·3 to + 0·1% for Cr and 0 to 2·5% for V.

A. G. COOPER

3711. **Methods of precipitation of hydrated oxides at constant pH. Principle and application to the micro-analysis of chromium and molybdenum.** Motoharu Tanaka (Fac. des Sci., Nagoya, Japan). *Mikrochim. Acta*, 1958, (2), 204–211 (in French).—The principle of pptg. oxides of Fe^{III} and Mn^{IV} at constant pH is given, and the importance of the pH value in co-pptn. is discussed. This method is shown to be effective in concentrating and separating a very small amount of an element. The behaviour of Mo and Cr in such a pptn. is discussed.

D. F. PHILLIPS

3712. **Paper electrochromatography: separation of chromium(III), chromate, molybdate and tungstate.** L. Blum. *Rev. Chim., Bucharest*, 1958, **9** (1), 28–34.—A horizontal electrochromatographic cell with two graduated scales was built to study the movement of coloured ions. The following conclusions were drawn. The displacement of the ions is proportional to time, and the possibility of separation increases with time, especially with higher potential gradients. Ionic migration is proportional to the voltage applied, the best separation being obtained with high voltages. The velocity of the ions is influenced by the position of the starting point with respect to the + ve or − ve electrodes. The speed of ions not adsorbed by the paper is independent of their concn., while the adsorbed ions form definite zones if in low concn. (0·1 *M*). The velocity of the ions studied is proportional to the concn. of the electrolyte, the best separation being effected with 0·1 *M* concn. of ammonium citrate and lactic acid. The ionic displacement is largely dependent on the electrolyte: the same ion can behave as cation, anion, or isoelectrically by change of electrolyte. Good separation of Cr^{3+} and CrO_4^{2-}; CrO_4^{2-} and MoO_4^{2-}; and CrO_4^{2-} and WO_4^{2-} is obtained with ammonium citrate (0·1 *M*); and of MoO_4^{2-} and WO_4^{2-} with lactic acid (0·1 *M*) as electrolytes, with a potential gradient of 7 V per cm. The time required is 30 to 60 min. Successful trials of a continuous separation of the same groups were made. H. SHER

3713. **Amperometric determination of molybdenum.** V. M. Peshkova, Z. A. Gallaĭ and N. N. Alekseeva. *Khimiya Redkikh Elementov*, 1957, (3), 119–130; *Ref. Zhur., Khim.*, 1958, Abstr. No. 7600.—A method is described for the amperometric titration of Mo^{VI} with a soln. of Cr^{2+}. The Mo^{VI} is quant. reduced to Mo^{V} in a basal soln. of 4 *N*

HCl, 4 *N* H_2SO_4 and 4 *N* H_3PO_4. A rotating platinum micro-electrode is used. No interference is caused by Mn, Zn, Al, Cr, Co or other metals. It is possible to determine Mo in the presence of Ti with a ratio of Mo to Ti of 1:4, and in the presence of W with a ratio of Mo to W of 1:15. It is established that it is possible to determine W^{VI} in pure soln. in a basal soln. of 7 *N* HCl. A method is also described for determining Mo in soln. containing Fe with a ratio of Fe to Mo of 10:1. With higher concn. of Fe, a chromatographic separation of Fe and Mo is carried out. The applicability of the amperometric method for the determination of Mo in ferromolybdenum and steel is indicated.

C. D. KOPKIN

3714. **Coulometric determination of molybdenum compounds. I. Determination of molybdenum and phosphorus.** Hiroshi Iinuma and Takayoshi Yoshimori. *Res. Rep. Fac. Eng., Gifu Univ., Japan*, 1957, (7), 66–70.—From 50 to 400 μg of Mo can be determined successfully as follows. Reduce the molybdenum compound with zinc amalgam to form Mo^{3+}, oxidise the Mo^{3+} with Fe^{3+} and determine the resulting Fe^{2+} by a coulometric oxidation. From 20 to 200 μg of P is indirectly determined as follows. Precipitate P as molybdophosphate by the addition of acid molybdate reagent, warm to 60°, and set the soln. aside overnight. Wash the ppt. with dil. NH_4NO_3 and HNO_3 soln., then dissolve it in NaOH soln. Add H_2SO_4 and remove HNO_3 by fuming. Determine Mo in the soln. as before, and P is calculated from the ratio P:Mo = 1:12. The ammonium molybdophosphate ppt. must be prepared under specified conditions. CHEM. ABSTR.

3715. **Amperometric determination of molybdenum and tungsten in the presence of nickel.** A. K. Babko and A. I. Volkova (Inst. of Gen. and Inorg. Chem., Acad. Sci., UkrSSR). *Zavod. Lab.*, 1958, **24** (2), 135–137.—When Ni is present in the amperometric titration at pH 6·5 to 7 of molybdate and tungstate with 0·5 *M* $Pb(NO_3)_2$, a complexing agent (glycine or ethylenediamine) is added to prevent the reaction of Ni with molybdate and tungstate. The hot soln. (80°) is titrated at a potential of 1 V *vs.* the S.C.E. by means of a polarograph. Titration can be carried out just as accurately without a polarograph if a suitable galvanic element is used. G. S. SMITH

3716. **Catechol violet as a colorimetric reagent for the determination of molybdenum, vanadium and tungsten.** A. K. Majumdar and C. P. Savariar (Jadavpur Univ., Calcutta, India). *Naturwissenschaften*, 1958, **45** (4), 84.—The dye forms coloured complexes at pH 2 to 4 with Mo^{VI}, V^{V} and W^{VI}. The brownish-blue complex of Mo has an absorption maximum at 530 mμ, the yellow complex of V at 420 mμ and the blue complex of W at 540 mμ. The colour reaction is highly dependent on pH, and anions such as fluoride, citrate and oxalate interfere. The complexes of Mo and V obey Beer's law at concn. of 1 to 10 p.p.m., that of W in the range 1 to 6 p.p.m. E. KAWERAU

3717. **Quantitative determination of tungsten.** S. I. Gusev, V. I. Kumov and R. G. Belles. *Trudy Molotovsk. Med. Inst.*, 1957, (26), 155–157; *Ref. Zhur., Khim.*, 1958, Abstr. No. 894.—The gravimetric determination of W with diantipyrinylphenylmethane (**I**) is described; the method is not less accurate than the cinchonine method and is considerably more rapid. In addition, the decomposition of ferrotungsten is accelerated. Dissolve the sample of ferrotungsten (≃ 0·25 g) in 25 ml of a saturated oxalic acid soln. with the addition of 4 ml of 30% H_2O_2 in 1-ml portions. To the soln. add conc. H_2SO_4 (10 ml) and 2% $KMnO_4$ soln. (30 to 40 ml), and destroy excess of $KMnO_4$ with one drop of H_2O_2 soln. Dilute the soln. to 150 ml, add HCl (sp. gr. 1·19) (10 ml), heat to b.p., add a 2% ethanolic soln. of **I** (4 ml), set aside for 2 hr., filter, wash the ppt. with 0·01% **I** soln. in 2% HCl, dry, char and ignite. C. D. KOPKIN

3718. **Separation of uranium from diverse ions—study of the hexone [*iso*butyl methyl ketone] liquid-liquid extraction system.** W. J. Maeck, G. L. Booman, M. C. Elliott and J. E. Rein (Phillips Petrol. Co., Idaho Falls, Ida.). *U.S. Atomic Energy Comm.*, Rep. IDO-14415, 1957, 22 pp.—A liquid-liquid extraction procedure is presented for the separation of U^{VI} from complex mixtures. One batch contact gives > 99·8% extraction of U^{VI}; < 1% of 1-yr. cooled fission products is extracted giving a procedure especially suitable for U^{VI} separation from reactor fuel element soln. prior to the determination of U^{VI}. The salting agent is 4 ml of 2·8 *M* $Al(NO_3)_3$ made 0·1% in tetrapropylammonium nitrate and *M* in NH_3. A 0·5-ml sample aliquot is added to the salting soln. and the U is extracted into 2 ml of *iso*butyl methyl ketone. Only a few other elements such as Hg, Tc, Ru, Pu, Np and Th are extracted to any extent. The effects of 30 anions on the extraction of U^{VI} and fission products have been established; only $Fe(CN)_6^{4-}$ and WO_4^{2-} hold back U. Five anions ($Cr_2O_7^{2-}$, I^-, $S_2O_3^{2-}$, SCN^- and WO_4^{2-}) slightly increase fission-product extraction.

CHEM. ABSTR.

3719. **A new volumetric method for the determination of uranium(VI). Photochemical reduction with ether.** V. Panduranga Rao and G. Gopala Rao (Andhra Univ., Waltair, India). *Z. anal. Chem.*, 1958, **160** (3), 190–193 (in English).—In soln. in *N* H_2SO_4 saturated with ether, U^{VI} is reduced to U^{IV} by exposure for 1 hr. to sunlight or to a high-pressure mercury-vapour lamp. The U^{IV} can be determined by titration with Na_3VO_4. Results are in good agreement with the amounts of U^{VI} taken. The reduction is inhibited by Br^- and Cl^-; F^-, PO_4^{3-}, AsO_4^{3-} and ClO_4^- do not interfere with the reduction or the titration. G. BURGER

3720. **Sodium alizarin-3-sulphonate [alizarin red S] as a colorimetric reagent for the micro-determination of uranium.** Anil K. Mukherji and Arun K. Dey (Univ. of Allahabad, India). *Z. anal. Chem.*, 1958, **160** (2), 98–104 (in English).—Uranium in soln. as the chloride, nitrate or sulphate in concn. of 40 to 250 p.p.m. can be determined by means of Na alizarin-3-sulphonate. A 20-fold excess of the reagent is added to the soln. at pH 5 ± 0·5, and the extinction is measured at 500 mμ at a temp. between 20° and 30°. Under these conditions Beer's law is followed and the colour is stable. Many ions interfere. The smallest concn. of U detectable is 10 p.p.m. G. BURGER

3721. **Fluoride method for separating small quantities of uranium for subsequent polarographic determination.** I. E. Starik, F. E. Starik and A. N. Apollonova. *Trudy Radiev. Inst., Akad. Nauk. SSSR*, 1956, **7**, 107–110; *Ref. Zhur., Khim.*, 1958, Abstr. No. 896.—The polarographic determination

of U, after reduction of U^{VI} with zinc and pptn. as UF_4 on a ThF_4 carrier, is described. Treat the sample with a mixture of HF and H_2SO_4, precipitate the sesquioxides with aq. NH_3 and dissolve the ppt. in 15% H_2SO_4 or HCl (50 ml). Reduce U^{VI} in the soln. with 20 g of pure granulated zinc for 20 min., filter into a platinum dish containing 30% NH_4F soln. (30 ml) and 40% HF (10 ml), add 0·5 *N* $Th(SO_4)_2$ (1 ml), stir, set aside for 1 hr. and filter through an ebonite funnel. Wash the ppt. with HF (1:1), treat it with a mixture of H_2SO_4 and HNO_3 in a platinum dish, evaporate to SO_3 fumes, dissolve the residue in water and precipitate the hydroxides of Th and U with aq. NH_3. Dissolve the ppt. in 10% HCl, evaporate, dissolve the residue in 0·1 to 0·5 *N* HCl and subject to polarography in an acid medium (0·1 to 0·5 *N* HCl - 0·3 to 0·5 *N* H_2SO_4). Satisfactory results are obtained for 10^{-4} to 10^{-5} g of U. No interference is caused by Fe, Al or V. The method may be used for the determination of U in rocks or water.

C. D. KOPKIN

3722. **Determination of uranium in UO_2 - Al_2O_3 fuel elements by X-ray emission spectrography.** W. D. Moak and W. J. Pojasek (Gen. Elect. Co., Schenectady, N.Y.). *U.S. Atomic Energy Comm.*, Rep. KAPL - 1879, 1957, 15 pp.—Sections of the fuel element are fused with $K_2S_2O_7$ followed by dissociation of the fused melt in water to which is added a few drops of H_2SO_4. The sample is excited by radiation from a tungsten tube. The U L_α emission line is diffracted by a LiF single crystal with *d*-spacing of 2·014 A, and the diffracted beam is detected by a scintillation counter. The coeff. of variation is ± 1·8%. CHEM. ABSTR.

3723. **[Determination of] uranium and barium in stone meteorites.** H. Hamaguchi, G. W. Reed and A. Turkevich (Enrico Fermi Inst. for Nuclear Studies, Univ. Chicago). *Geochim. et Cosmoch. Acta*, 1957, **12** (4), 337–347.—A radioactivation procedure involves thermal neutron irradiation of samples of meteorite along with a monitor of meteoritic composition but spiked with microgram quantities of U. After irradiation, the sample and monitor are dissolved in the presence of ^{237}Np and Ba carrier: the former serves as a tracer for 2·33-day ^{239}Np produced by neutron capture of ^{238}U and subsequent β-decay, and the latter as a carrier for 12·8-day ^{140}Ba produced by ^{235}U fission. The amount of U in the meteorite is then determined by comparing the ^{239}Np and ^{140}Ba - ^{140}La activities isolated from the samples with those from the monitor: then, assuming normal isotopic composition for the U present, two independent determinations of the U content are given. By using the assumption of terrestrial isotopic composition for the Ba also, the Ba isolated after irradiation contains, besides ^{140}Ba, radioactive species induced by the interaction of neutrons with Ba present in the meteorite, which can be used to estimate the Ba contents of the samples. R. A. HOWIE

3724. **New method for enriching uranium by means of ion exchange and its use in the determination of uranium in solid samples.** J. Korkisch, A. Farag and F. Hecht (University Chem. Inst., Vienna). *Mikrochim. Acta*, 1958, (3), 415–425.—A soln. of a uranium compound in HCl reacts with ascorbic acid at pH 4·0 to 4·5 to give a stable complex, which is strongly adsorbed on the basic ion-exchange resin Amberlite IRA-400 and can be eluted with *N* HCl. The U in the eluates is determined polarographically as the nitrate. By this procedure, both microgram and milligram amounts of U can be completely separated from alkali metals, alkaline-earth metals, Al, Pb, As^{3+}, Bi^{3+}, Zn, the rare earths, Cr^{3+}, Mn^{2+}, Fe^{2+}, Co and Ni. Sulphate and chloride and small amounts of phosphate or fluoride do not interfere.

S. M. MARSH

3725. **Isotope dilution: α-spectrometer for uranium and thorium determination.** L. E. Howard (Bureau of Mineral Resources, Melbourne, Australia). *Nucleonics*, 1958, **16** (2), 112–121.—In the determination of U in Th-free minerals, exchange is established between the ^{230}Th in the sample and added ^{232}Th. The Th is then extracted, not necessarily completely, purified, and examined in a gridded ion chamber; the concn. of U is deduced from the intensities of the ^{230}Th and ^{232}Th peaks. The determination of Th in U-free minerals is similar, the standard nuclide used is ^{232}Th and it is added in pitchblende in which the ^{231}Th is in equilibrium with the ^{235}U. If the mineral contains Th and U it can be examined directly to determine the ^{230}Th to ^{232}Th ratio so that corrections may be applied to the previous procedures; alternatively, a mixture of ^{228}Th and ^{232}Th which has a different ^{228}Th to ^{232}Th ratio from that in the mineral is added, and the contents of U and Th are deduced from the intensities of the ^{228}Th, ^{230}Th and ^{232}Th peaks. Minerals containing as little as 0·1% of U or Th have been analysed. G. J. HUNTER

3726. **Emission and absorption spectra of plutonium excited in a King furnace.** L. Bovey (A.E.R.E., Harwell, England). *Spectrochim. Acta*, 1958, **10** (4), 383–394.—A list is given of wavelengths and relative intensities (estimated visually) of the absorption and emission lines of Pu excited in a King-type furnace (*cf. Anal. Abstr.*, 1956, **3**, 884), and photographed over the range from 2791 to 6888 A. An overall accuracy of ≃ 0·05 A is claimed.

A. R. ROGERS

3727. **Determination of the ratio of plutonium to uranium in pitchblende.** I. E. Starik, A. P. Ratner, M. A. Pasvik and F. L. Ginzburg. *Geokhimiya*, 1957, (2), 142–146; *Ref. Zhur., Khim.*, 1958, Abstr. No. 7591.—A study is made with radioactive tracers of methods of separation of Pu from Pa, Ra, Po and isotopes of Th by co-pptn. of Pu with uranyl acetate and extraction with diethyl ether. The results obtained are used for working out a procedure for separating Pu from pitchblende by a combination of the two methods. From 2 kg of pitchblende containing 43·5% of U, ^{239}Pu is obtained with a chemical yield of 33·3%. As a control for the chemical yield of ^{239}Pu in the ore soln., ^{238}Pu is introduced as tracer. Identification by the energy of the α-particles shows that, in the separated sample containing ^{238}Pu and ^{239}Pu, other α-sources are absent (accuracy 2·5%). It is established that the ratio of Pu to U in the pitchblende studied is $(2 \pm 0{\cdot}3) \times 10^{-11}$. C. D. KOPKIN

3728. **Separation of halogenates by ion exchangers.** M. Kikindai-Cassel. *Ann. Chim., Paris*, 1958, **3** (1–2), 5–51 (in French).—The ions ClO_3^-, BrO_3^- and IO_3^- are separated by ion-exchange chromatography on a column (50 cm × 1 cm) of a strongly basic resin (Dowex 2). Elution is effected by KOH soln. for IO_3^- and BrO_3^-, and K_2SO_4 soln. for ClO_3^-, the order for elution from the column being $IO_3^- > BrO_3^- > OH^- > ClO_3^-$. A good separation is

achieved with a total of 4·5 milli-equiv. on a column of 23 theoretical plates. The test soln. ($\simeq$ 10 ml) is allowed to run through the column at $\simeq$ 0·3 ml per min., and the eluting soln. (0·2 *N* KOH and 0·1 *N* K_2SO_4) at $\simeq$ 0·20 ml per sec.

J. H. WATON

3729. **Colorimetric determination of fluorine.** A. A. Guntz and M. Arène. *Chim. Anal.*, 1958, **40** (2), 39–42.—To an acid soln. containing $<$ 10 μg of fluoride, prepared as described previously (*cf. Ibid.*, 1957, **39**, 260), add a fixed vol. of alizarin red S soln. to give a concn. of 20 to 60 mg per litre and chloroacetate buffer of pH 2·9. Then either add a fixed quantity of thorium soln. and measure the extinction at 530 mμ, or titrate with thorium soln. to give a fixed extinction. Alternatively, add a fixed vol. of thorium soln. to the sample and to a similarly prepared blank soln., and titrate the latter with fluoride soln. to an identical extinction.

A. R. ROGERS

3730. **Colorimetric determination of the fluoride ion based on interference with extractability.** Z. G. Szabó, M. T. Beck and K. Tóth (Szeged Univ., Hungary). *Mikrochim. Acta*, 1958, (2), 181–185 (in English).—The sensitivity of colorimetric determinations based on the fading produced by complex-forming anions can be increased by measuring the change of absorbancy in the organic phase after shaking with a suitable solvent, *i.e.*, by determining the interference of the anion with extractability. The ferric thiocyanate - fluoride system was examined. *Procedure*—Place 5 ml of $2·5 \times 10^{-4}$ *M* $(NH_4)_2SO_4.Fe_2(SO_4)_3$ and then 8 ml of 0·1 *M* NaSCN into a 50-ml flask. Add carefully neutralised fluoride soln. equiv. to 10 to 100 μg of F^-. Dilute to almost 50 ml and adjust the pH to exactly 2·9. Adjust the vol. to 50 ml and shake vigorously in a separating funnel with 15 ml of *n*-pentanol. Centrifuge to promote separation of the phases, and measure the extinction of the *n*-pentanol phase with an S50 filter in a Pulfrich photometer. Run a blank concurrently. Determine F^- from the extinction difference, by reference to a calibration curve. The accuracy is $\pm$ 5%. Determine fluorosilicate in same way. [*Cf. Magyar Kém. Foly.*, 1958, **64** (1), 35–37 (in Hungarian).] D. F. PHILLIPS

3731. **The titrimetric determination of bromide ions.** L. Szekeres (Univ. für landwirtschaftliche Wissenschaften, Budapest). *Z. anal. Chem.*, 1958, **160** (3), 198–201.—The Br^- soln. is treated at room temp. with excess of $KBrO_3$ in *N* H_2SO_4. After 15 min. the soln. is made alkaline with NaOH. The NaOBr formed is reduced with H_2O_2 and the excess of H_2O_2 is destroyed by heating. The excess of $KBrO_3$ is then determined by addition of KI and HCl and titration with $Na_2S_2O_3$. The vol. of titrant used is within 0·1 ml of the theoretical, even in the presence of a 30-fold excess of Cl^-. G. BURGER

3732. **Spectrographic determination of iodine in solutions.** S. M. Kishko and V. S. Shevera. *Nauch. Zap. Uzhgorodsk. Univ.*, 1957, **18**, 91–93; *Ref. Zhur., Khim.*, 1958, Abstr. No. 7633.—The iodine spectrum is excited in a low-voltage spark discharge between copper electrodes at a capacity of 100 μF. Introduce the soln. into a hollow in the lower electrode and photograph the spectrum with a medium quartz spectrograph and panchromatic plates. The analytical line is I 5875 A, with slit-width 10 μ and exposure 15 sec. The comparison line, current strength and inductance of the discharge circuit are not given. C. D. KOPKIN

3733. **The anion-exchange separation of iodine anions.** M. L. Good, M. B. Purdy and T. Hoering (Dept. of Chem., Louisiana State Univ., Baton Rouge, U.S.A.). *J. Inorg. Nuclear Chem.*, 1958, **6** (1), 73–75.—Iodide, IO_3^- and IO_4^-, labelled with ^{131}I, were eluted from a column (12 mm diam. by 50 cm height) of Amberlite IRA-400 (OH^- form), 20 to 50 mesh, first with 200 ml of NaOH soln. and then with one litre of *M* KNO_3; both soln. had a pH value of 10·5. Iodate and IO_4^- were recovered in the 25th to 70th-ml portion of the KNO_3 soln. and the I^- in the 300th to 1000th-ml portion.

G. J. HUNTER

3734. **Potentiometric micro-determination of periodate by arsenite - iodine titration.** A. F. Reid (Nat. Univ., Canberra, Australia). *Mikrochim. Acta*, 1958, (2), 236–240 (in English).—By following the iodine - arsenite titration potentiometrically, the Fleury - Lange method (*J. Pharm. Chim.*, 1933, **17**, 196) may be extended to the determination of 0·5 μmole of periodate with a precision of 1%, and 0·05 μmole with a precision of 3%. The excess of arsenite is titrated with dil. iodine soln.

D. F. PHILLIPS

3735. **Determination of manganese with persulphate in the presence of various catalysts.** N. P. Gaĭdarzhi. *Trudy Odessk. Univ.*, 1956, **146**, *Sb. Stud. Rabot*, (4), 129–132; *Ref. Zhur., Khim.*, 1958, Abstr. No. 899.—Study is made of the catalytic action of Co, Ni, Cu and Hg and their mixtures on the oxidation of Mn^{II} to Mn^{VII} with $(NH_4)_2S_2O_8$ (**I**). Best results are obtained with $CoSO_4$, with an error of $<$ 2% of Mn. At a low acidity of the soln. low results are obtained, because of separation of $Mn(OH)_4$. At high acidity the colour due to Mn^{VII} does not appear. The determination of the end-point of the titration of Mn^{VII} is rendered difficult by the pink colour of salts of Co^{II}. This disadvantage is overcome by the addition of salts of Ni^{II} in double or treble the amount of the cobalt salts added. In determining Mn^{II} by oxidation with **I** it is recommended to use mixed catalysts (Co and Ni; Co and Cu) instead of $AgNO_3$.

C. D. KOPKIN

3736. **Rapid polarographic determination of manganese in ferromanganese and manganese slag.** Tsurumatsu Dono, Ken-ichi Morinaga, Tsuyoshi Nomura and Genkichi Nakagawa (Nagoya Inst. of Tech., Showa-ku, Nagoya). *Japan Analyst*, 1958, **7** (1), 25–28.—The polarography of Mn^{III} in $Na_4P_2O_7$ ($E_{\frac{1}{2}} = +0·15$ V *vs.* the S.C.E.) (*cf.* Kolthoff and Watters, *Ind. Eng. Chem., Anal. Ed.*, 1943, **15**, 8) was applied to the analysis of ferromanganese (60 to 80% of Mn) and manganese slag ($<$ 20% of Mn). The wave remains unchanged at pH 2·5 to 3·4 and its height is proportional to the concn. of Mn for $<$ 50 mg per 100 ml in the presence of 0·01% of gelatin. The presence of Cl^-, Cr, V and a large amount of Fe ($>$ 5 times the wt. of the Mn) causes interference. The error is $<$ 1% relative. *Procedure*—Dissolve the sample containing 10 to 50 mg of Mn in conc. HNO_3 (15 ml) (or when insol. in HNO_3, decompose the sample with aqua regia and evaporate with HNO_3), dilute to $\simeq$ 10 ml, add $Na_4P_2O_7$ (13·5 g), adjust the pH to between 2·3 and 2·6 with NaOH or HNO_3 and make up to 100 ml with gelatin soln. (0·1%, 10 ml). Add PbO_2 (1 g), filter after 10 min. and polarograph.

K. SAITO

3737. **Determination of manganese in silicomanganese by surface testing.** G. A. Medvedeva. *Trudy Ural'sk. Politekh. Inst.*, 1957, (69), 129–131; *Ref. Zhur., Khim.*, 1958, Abstr. No. 21,178.—Two variations of the determination of Mn in silicomanganese by surface treatment are described. (*i*)—On the cleaned surface of the sample and on standards construct paraffin-wax cells of diam. 5 to 8 mm, into each of which introduce two drops of HF. After dissolution (5 to 7 min.) add two or three drops of water and transfer the soln. by a capillary pipette to a 100-ml flask containing 50 ml of water. Wash the surfaces of the samples with water and add the washings to the corresponding flasks. Make up the soln. to vol., mix, and to 2 ml of each soln. add five drops of HNO_3 (1:1), five drops of 1% $AgNO_3$ soln. and a speck of dry $(NH_4)_2S_2O_8$, heat and measure the extinction on an Eggertz colorimeter on a background of white frosted glass. (*ii*)—Into each of two 500-ml flasks add water (300 ml), HNO_3 (sp. gr. 1·40, 50 ml) and 1% $AgNO_3$ soln. (10 ml) and heat to boiling. During the heating place two drops of HF in paraffin-wax cells on the surface of the sample and standard. After dissolution, transfer the HF soln. to the flasks. Then add to each flask 25% $(NH_4)_2S_2O_8$ soln. (50 ml), heat to boiling, cool in running water and titrate with 0·03 *N* Na_3AsO_3. The abs. error in determining Mn by methods (*i*) and (*ii*) is, respectively, 3 and 1·5%.

C. D. KOPKIN

3738. **Polarographic determination of traces of iron in reagent-grade sodium chloride and caustic alkali.** Kishiro Sugihara and Toshihide Saito (Osaka Ind. Res. Inst., Ooyodo-ku, Osaka). *Japan Analyst*, 1958, **7** (1), 1–4.—A small amount of Fe (0·00005 to 0·0015%) in pure sodium chloride, potassium hydroxide or sodium hydroxide is co-pptd. with $Al(OH)_3$ and determined polarographically in an NaOH soln. containing mannitol (Komarek, *Coll. Czech. Chem. Commun.*, 1935, **7**, 407; Perkin and Reynolds, *Analyst*, 1953, **78**, 480). The co-pptn. of Fe (30 to 150 μg) is quant. when 8 mg of Al in < 300 ml of 30% NaCl (or KCl) soln. is used. *Procedure*—Prepare a 30% soln. of sodium chloride, add conc. HNO_3 (a few drops) and aluminium soln. ($\simeq$ 8 mg of Al, 1 ml) and boil with aq. NH_3 soln. Wash the ppt. with NH_4Cl soln. (2%), dissolve it in hot HNO_3 and evaporate to < 2 ml. Make up the product to 10 ml with mannitol soln. (0·4 *M*, 2·5 ml) and KOH soln. (8 *M*, 2·5 ml), record the polarogram and measure the limiting current of the wave due to Fe (− 1·18 V *vs.* the S.C.E.).

K. SAITO

3739. **Rapid determination of iron in brine.** Shizo Hirano and Eiitsu Igami (Inst. of Techno-anal. Chem., Fac. of Engng, Tokyo Univ., Hongo). *Japan Analyst*, 1958, **7** (2), 106–107.—Iron (< 500 μg) in brine (300 ml) is co-pptd. with MnO_2 and photometrically determined with 1:10-phenanthroline (**I**). No interference results from Cu, Zn, Ni, Cr, Ca, Mg or Ag; Hg is removed by washing the MnO_2 with KI soln. *Procedure*—Boil the sample (200 ml) with HCl (1 + 9, 1 ml), aq. Br soln. (1 ml), $MnSO_4$ soln. (4%, 10 ml) and water (total 400 ml) for several hours, make ammoniacal, add Na_2CO_3 (0·2 g) and aq. Br soln. (1 ml), and filter off the ppt. Wash the ppt. with water and KI soln. (5%, 10 ml), then dissolve it in HCl (1 + 9, 10 ml) and hydroxylamine hydrochloride soln. (10%, 1 ml). Make up this soln. to 100 ml with Na acetate (2 *M*, to adjust the pH to 3·5) and **I** (0·2%, 2·5 ml) and measure its extinction at 500 mμ after 10 min.

K. SAITO

3740. **Phthalocyanines as oxidation - reduction indicators. I. Use of copper phthalocyaninetetrasulphonic acid (Cu - PTS). Cerimetric determination of iron(II) and ferrocyanide.** G. Gopala Rao and T. P. Sastri (Andhra Univ., Waltair, India). *Z. anal. Chem.*, 1958, **160** (2), 109–114 (in English).—A 0·1% aq. soln. of Cu - PTS can be used as indicator in the volumetric determination of Fe^{2+} and of $Fe(CN)_6^{4-}$ in acid soln. with $Ce(SO_4)_2$. The end-point, shown by the appearance of a transient pink colour, is sharp even with a titrant as dilute as 0·001 *N*. No interference is caused by HgCl, $HgCl_2$, $SnCl_4$, WO_4^{2-} or As^{3+}.

G. BURGER

3741. **Use of anion-exchange resins in analytical chemistry. IV. Separation of iron and aluminium on the anion-exchange resin OAL.** I. Kulčickyj and F. Švácha (Závod První Pětiletky, Šumperk, Czechoslovakia). *Chem. Listy*, 1958, **52** (2), 340–342.—Iron in strongly acid medium forms the complex anion $FeCl_4^-$ which can be retained on a strongly alkaline anion-exchange resin; aluminium does not react in this manner and can thus be separated. *Procedure*—Dilute a mixture of iron and aluminium salts with conc. HCl to 25 ml and transfer to an OAL column. Wash with conc. HCl (50 ml) (at 2·5 ml per min.) and determine Al in the eluate gravimetrically as Al_2O_3. Elute the Fe from the column with 0·1 *N* HCl (50 ml) (at 5 ml per min.) and determine Fe in the eluate gravimetrically or by iodimetric titration. The complete analysis does not take more than 1 hr. The method can also be used for the determination of Fe in technical HCl and for the purification of this acid. For technical purposes a column of diameter 100 mm containing 5000 cu. cm of OAL was used.

J. ZÝKA

3742. **Rapid photometric micro-determination of iron in chromium, manganese and nickel by the oxinate extraction method.** Kenji Motojima and Hiroshi Hashitani (Japan Atomic Energy Res. Inst., Tokai, Ibaragi). *Japan Analyst*, 1958, **7** (1), 28–33.—Since the Fe - 8-hydroxyquinoline complex (max. absorption, 470 and 580 mμ) is extracted with $CHCl_3$ at pH < 2·4, Fe (< 100 μg) can be separated from Mn (< 150 mg), Ni (< 120 mg) and Cr (< 100 mg) and determined photometrically at 580 mμ. The extinction coeff. of the 8-hydroxyquinoline complexes of Cu, Mo and W, which are extracted together with Fe, are negligible at 580 mμ. Vanadium (> 0·5 mg) interferes, and in its presence (< 0·5 mg) the $CHCl_3$ soln. is washed with 0·1 *N* acetic acid and basic ammonium buffer (Motojima, *Anal. Abstr.*, 1958, **5**, 463). An acid soln. (35 ml) of the sample (metallic nickel, chromium or manganese containing < 0·01% of Fe) is mixed with the reagent (1% in acetic acid, 3 ml) adjusted to a pH between 2·5 and 3·3 with ammonium acetate and aq. NH_3 soln., diluted to 50 ml and extracted with $CHCl_3$ (10 ml).

K. SAITO

3743. **Determination of ferrous and ferric oxide in an iron catalyst.** Yoshio Murata and Shigeaki Kasaoka (Fac. of Educ., Univ. of Osaka Prefecture, Sakai). *Japan Analyst*, 1958, **7** (1), 50–52.—After the metallic iron in an iron catalyst ($\simeq$ 0·5 g) is separated by the $HgCl_2$ method (*Anal. Abstr.*, 1956, **3**, 3049), the residue is heated with ethanolic iodine (4%, 50 ml) (to remove the metallic Hg) for 30 min.

under reflux, and filtered off. The residue is washed with ethanol, dissolved in H_2SO_4 (1 + 10, 100 ml) in a current of CO_2 and titrated with $KMnO_4$ (0·02 to 0·1 *M*). The product is reduced with aluminium (≃ 0·5 g), filtered and again titrated with $KMnO_4$ (for the sum of FeO and Fe_2O_3). K. SAITO

3744. **Determination of FeO, MnO and of dissolved gases in iron.** M. Šícha (Vítkovické Železárny, Ostrava, Czechoslovakia). *Hutn. Listy*, 1958, **13** (2), 185–192.—In a re-investigation of the electrolytic isolation of free FeO and MnO in steel it was found that it is not possible to separate both oxides quantitatively. When decomposing the electrolytic residue by chlorination in a vacuum apparatus, considerable losses were observed. By the use of the method of fractional vacuum extraction with the addition to the sample of Sn or graphite, respectively, the total amount of both oxides can be determined. In gases, liberated from grey pig iron and from cast iron by heating the sample to 600° in vacuum, the content of methane in which 45% on the average of the total hydrogen is bound was analytically determined. J. ZÝKA

3745. **Quantitative determination of ferrates by the potassium iodide oxidation reaction. Preliminary communication.** N. M. Mezentseva, R. L. Slutskin and V. N. Fedorushin. *Sb. Stud. Nauchno-Issled. Rabot. Sibirsk. Lesotekhn. Inst., Krasnoyarsk*, 1957, **3**–7; *Ref. Zhur., Khim.*, 1958, Abstr. No. 11,004.—A method is described for the titrimetric determination of FeO_4^{2-}, based on the reaction—

$$K_2FeO_4 + 4KI + 2H_2SO_4 \rightarrow K_2SO_4 + 2I_2 + FeSO_4.$$

To 10 ml of K_2FeO_4 soln. add an excess of KI soln. (1·5%), mix (in an atmosphere of CO_2) with a soln. of 7 g of KI and 5 ml of H_2SO_4 (1 : 1) in 100 ml of H_2O, set the mixture aside in the dark for 30 min. and titrate the liberated iodine with $Na_2S_2O_3$ soln. in an atmosphere of CO_2. The results obtained are close to those of a method described previously (Petrov and Ormont, *Zhur. Obshch. Khim.*, 1937, **7**, 1690) but are somewhat lower. It is established that, in the described reaction, spontaneous decomposition of ferrate does not occur.

C. D. KOPKIN

3746. **Analysis of residues from pig and cast iron.** A. Wittmoser, H. Bockshammer and W. D. Gras (Inst. Met. Research, Gelsenkirchen, Germany). *Rev. Mét.*, 1957, **54**, 369–382; 419–432.—In contrast with the conventional electrolytic methods, the authors attempted complete dissolution of the disc-shaped specimen as anode (40 mm diam., 1 mm thick). The electrolyte is comprised of a soln. containing 5% of Na citrate, 0·025% of dibenzyl sulphoxide, 0·05% of a sulphonate wetting agent, and 0·1% of NaF. A magnetic separator and a method of flotation are described for the separation of the compounds in the residue. The magnetic separator consists of 2 parts—(*i*) a beaker containing an ethanolic suspension of the isolated constituents and a rotating magnetic stirrer outside the beaker, and (*ii*) a second, smaller beaker containing a magnet which is immersed from the top into the suspension. The movement of the magnetic stirrer separates locally the graphite and non-magnetic particles from cementite. The cementite particles adhere to the bottom of the second beaker and are removed at intervals. A separation by flotation follows to separate the non-magnetic constituents, *e.g.*, graphite, oxides, sulphides, phosphates and nitrides. The total losses that occur during the separation are of the order of 5%, which lies within acceptable limits of analytical technique.

CHEM. ABSTR.

3747. **New procedure for sampling killed and rimming molten steels for the determination of oxygen and hydrogen.** H. Schenck, K. H. Gerdom and K.-G. Schmitz (Rhein-Westf. Tech. Hochschule, Aachen). *Arch. Eisenhüttenw.*, 1957, **28** (3), 123–125.—The sample is taken by means of an evacuated quartz pipette fitted with a plug of fusible iron in a narrow neck at one end. As soon as the sample has been taken, the pipette is placed in a water-cooled tube from which the air can be swept by means of mercury. This tube is fitted with a vessel for collecting gas evolved during the cooling of the sample. When cool, the pipette is transferred to another vessel and cooled to — 100° until the usual procedure for the determination of the remaining gases can be carried out. In this way loss of hydrogen is prevented. G. BURGER

3748. **Influence of arsenic on the analysis of iron and steel. I. Determination of sulphur and phosphorus in iron and steel.** Hidehiro Gotô and Shiro Watanabe (Tôhoku Univ., Sendai). *Sci. Rep. Res. Insts, Tôhoku Univ., A*, 1956, **8**, 157–165.—The effects of As on the determinations of S and P were studied and a method for avoiding the interference of As is described. Sulphur was determined volumetrically by ignition to SO_2 and SO_3, by dissolution in non-oxidative strong acids to form H_2S, which is then determined volumetrically by iodine, and gravimetrically by pptn. as $BaSO_4$. In each of these three methods the presence of As has no effect on the accuracy of the determination of S. It is suggested that an efficient absorbing bottle of $KMnO_4$ soln. be used in the second method to reduce the health hazard from AsH_3. Phosphorus is determined by standard methods by alkalimetric titration after ammonium molybdate pptn. Arsenic interferes by forming a yellow ppt. of ammonium molybdoarsenate and is usually removed as a sulphide or bromide. Data are presented which show that the effect of As on P can be removed by carrying out the pptn. at lower temp. (30° to 40°).

II. Determination of copper and manganese in iron and steel. Hidehiro Gotô and Shiro Watanabe. *Ibid.*, 1956, **8**, 223–229.—The influence of As on the determination of Cu and Mn in iron and steel was studied. There was no direct influence of As on the determination of Cu by the $Na_2S_2O_3$ separation method and H_2S separation method. In the separation of Cu from Fe with H_2S, the amount of Fe co-pptd. with sulphides became larger in the presence of As, and re-pptn. of $Fe(OH)_3$ was necessary for the complete separation of Cu. Arsenic has no effect on the determination of Mn by any of the usual methods. CHEM. ABSTR.

3749. **Rapid spectrophotometric determination of zirconium in steel with quinalizarin.** Shigeo Wakamatsu (Toto Seiko Co., Minami-sunamachi, Koto-ku, Tokyo). *Japan Analyst*, 1958, **7** (2), 84–88.—Almost all ions that interfere with the colorimetric determination of Zr with quinalizarin (**I**) (Liebhapsky and Winslow, *J. Amer. Chem. Soc.*, 1938, **60**, 1776), including those of Fe^{II}, Cu, Al, Ni and Zn, are masked with EDTA at pH 5. Titanium and a large amount of Cu (> 1% in steel), Mo (> 1%), V (> 1%), Co (> 0·5%) and Cr (> 0·3%) cause interference. The working curve is linear for < 0·4 mg of Zr per 100 ml, and the error is

< 0·01% (absolute) for > 0·01% of Zr. *Procedure*—Dissolve the sample (0·1 g) in HCl (1 + 3, 15 ml) and H_2O_2 (30%, 3 ml). Partly neutralise with aq. NH_3 soln. and boil with Na_2SO_3 (1 g) for a few minutes. Add EDTA (disodium salt, 1 g), HCl (1 + 1, 4 ml), ammonium acetate soln. (50%, 16 ml), **I** (0·01% in ethanol) (20 ml) and ethanol (10 ml) and adjust the pH to 5·0. Heat the soln. at 80° to 90° for 5 min., cool, make up to 100 ml and measure the extinction at 540 mμ. A determination takes less than 25 min. K. SAITO

3750. **Colorimetric determination of titanium in alloy steel by the method of additions.** A. I. Lazarev and V. I. Lazareva (Industrial Inst., Kuibishev). *Zavod. Lab.*, 1958, **24** (2), 145–146.—The method is based on the use of three equal aliquots of the soln. to be analysed. The first is treated with water only and serves as a blank, the second is treated with H_2O_2 and water, and the third is treated with a known amount of $Ti(SO_4)_2$ soln. and also H_2O_2 and water. From the extinctions and the fact that the Beer - Lambert law is obeyed, the content of Ti can be calculated. To determine Ti in steel, the sample (0·25 g) is dissolved in 50 ml of dil. H_2SO_4 (1:4) and the soln. is treated with a few drops of conc. HNO_3. After removal of oxides of N, the cooled soln. is diluted to 100 ml and three 25-ml aliquots are taken. To two of them are added 2 ml of 3% H_2O_2 soln. and one of these is treated, in addition, with a soln. containing 0·05 to 1 mg of Ti as sulphate. Each of the three aliquots is then treated with 3 ml of H_3PO_4 (sp. gr. 1·70) and diluted with water to 50 ml. In measuring the extinction, a 400-mμ filter is used. The method has been proved satisfactory with alloys containing from 0·09 to 0·67% of Ti. G. S. SMITH

3751. **Volumetric determination of small amounts of cobalt with a ratio of equivalent weights of 1:37.** L. Bartha and S. Görög (Inst. for Inorg. and Anal. Chem., Sci. Univ. of Szeged, Hungary). *Magyar Kém. Foly.*, 1958, **64** (2), 48–50.—Cobalt is pptd. as $KPb[Co(NO_2)_6]$ and the ppt. is decomposed with alkali. The NO_2^- are reduced with a ferrous salt and the excess of the reducing agent is back-titrated. For the reduction of the six NO_2^-, 36 equiv. of Fe^{2+} are required, and one further equiv. is needed for the reduction of Co^{III}; thus the equiv. wt. of Co^{III} is one thirty-seventh of the atomic wt. *Procedure*—For the determination of 0·3 to 3 mg of Co, add the approx. neutral soln. of the cobalt salt (5 ml) to a centrifuge tube. If SO_4^{2-} are absent, add K_2SO_4 until the proportion of Co to SO_4^{2-} is 1:1 to 4 (in equiv. wt.). Add acetic acid (0·3 ml), 18% Pb acetate soln. (1 ml) and saturated KNO_2 soln. (1 ml). Set the mixture aside for 1 hr., then centrifuge it for 20 min. at 3000 r.p.m. Wash the ppt. with saturated K_2SO_4 soln. (2 × 3 ml), centrifuging each time for 10 min. Transfer the ppt. with H_2O (90 ml) to a 250-ml flask, add 30% NaOH soln. (10 ml) and boil for 10 min. Then to the boiling soln. add 19% $FeSO_4$ soln. (10 ml, containing 30 ml of conc. H_2SO_4 per litre). Put a mouthpiece with a long tube on the flask. After 3 to 6 min., carefully add conc. H_2SO_4 through the attachment and boil until the ppt. dissolves. Titrate the unoxidised ferrous salt in the cold with 0·1 *N* $KMnO_4$. A blank is carried out similarly, and the difference in the $KMnO_4$ consumption is a measure of the Co. If 30 to 300 μg of Co is present, use the following quantities—unknown soln., 2 ml; acetic acid, 0·2 ml; Pb acetate and KNO_2 soln., 0·5 ml each; proportion of Co and SO_4^{2-}, as above. After 1 hr. centrifuge the mixture for 10 min.; wash the ppt. with K_2SO_4 soln. (3 × 3 ml), centrifuging and washing for 5 min. Wash the ppt. with water (25 ml) into a 100-ml flask; add 30% NaOH soln. (5 ml) and boil for 5 min. Then add $FeSO_4$ soln. (5 ml), boil again for 5 min. and add dropwise 4 *N* HCl (20 ml). Stop the boiling and add immediately a few pieces of marble or $CaCO_3$. Cool the soln. to between 40° and 50°, add KI (5 g) and titrate with 0·01 *N* $Na_2S_2O_3$. A blank must be carried out. With 0·1 *N* soln. the error is ± 1%; on the micro scale, it is ± 5%; as little as 40 μg of Co can be determined, but the error is greater. A. G. PETO

3752. **Amperometric titration of cobalt with potassium ferricyanide by means of a rotating platinum micro-electrode.** A. K. Zhdanov, V. A. Khadeev and G. P. Moiseeva (V. I. Lenin Central Asiatic State Univ.). *Zavod. Lab.*, 1958, **24** (2), 137–140.—With a rotating platinum electrode and a S.C.E. (as anode), accurate amperometric titrations of Co in ammoniacal soln. containing ammonium salts can be carried out with $K_3Fe(CN)_6$ soln. No interference is caused by SO_4^{2-}, NO_3^-, Cl^-, acetate, citrate and tartrate, and the addition of citrate or tartrate prevents the interference of Ni, Zn, Cd, Pb, Bi, Cu, Fe and Cr. G. S. SMITH

3753. **Amperometric determination of cobalt in magnet alloys.** V. F. Toropova and O. S. Strekalova. *Zavod. Lab.*, 1958, **24** (2), 140–141.—Addition of $K_3Fe(CN)_6$ followed by amperometric titration of the excess with 0·1 *M* $CoSO_4$ at − 0·2 V *vs.* the S.C.E. is recommended. Tartrate or citrate is added to combine with Fe and Al. There is no interference from Ni. Copper is removed electrolytically or by pptn. as sulphide. G. S. SMITH

3754. **The collector in analytical chemistry. I. Colorimetric determination of traces of cobalt by the use of aluminium hydroxide as collector.** Teruo Imai and Kunio Seto (Fac. of Sci., Tohoku Univ., Katabira-cho, Sendai). *Japan Analyst*, 1958, **7** (1), 4–8.—Co-pptn. of Co (≃ 10 μg) with $Al(OH)_3$ (Okác and Bezdék, *Chem. Listy*, 1950, **44**, 300) is best carried out at pH 7·4 to 9·0, and the ppt. coagulates well on digestion at 30° for 15 min. Since NH_4^+ interfere with the subsequent colorimetric analysis of Co with nitroso-R salt, NaOH is preferred as a precipitant. The cobalt soln. is mixed with a soln. of Al (20 mg, 1 ml) and the pH is adjusted with 0·2 *N* NaOH. The presence of ethanol decreases the ratio of co-pptn.

K. SAITO

3755. **Vanadatometric micro-determination of nickel.** A. A. Verdi-Zade. *Trudy Azerb. Gos. Zaochn. Ped. Inst., Azerb. Devlet Giyabi Ped. Inst. Eserleri*, 1957, **4** (1), 77–81; *Ref. Zhur., Khim.*, 1958, Abstr. No. 7608.—Results are described of the qual. detection and quant. (gravimetric and titrimetric) determination of Ni with the use of hexavanadic acid (**I**). It is established that the reaction of $[Ni(NH_3)_4]^{2+}$ with **I** in the presence of ammonium salts produces the complex compound $[Ni(NH_3)_4]V_6O_{16}$ or a mixture of $[Ni(NH_3)_4]V_6O_{16}$ and $[Ni(NH_3)_6]V_6O_{16}$. The physical properties of the compound obtained are studied. The detectable minimum is 6 μg of Ni per ml. For the gravimetric determination of Ni, to 1 ml of soln. add 0·3 ml of a saturated ammonium salt soln., a soln. of **I** to a concn. of 0·03 to 0·035 *N* and 2 to 2·5 ml of 20% aq. NH_3. After 3 or 4 min. filter, wash the ppt. 5

or 6 times with 67% ethanol, ignite to between 500° and 580° and weigh as $NiO.3V_2O_5$. In the titrimetric determination, dissolve the complex nickel ppt. in 20 to 25 ml of H_2SO_4 (1:6) and titrate with a soln. of $(NH_4)_2SO_4.FeSO_4$ in the presence of phenylanthranilic acid. The results are satisfactory. C. D. KOPKIN

3756. **Analytical reactions of some amidoximes. VIII. Polarographic determination of nickel with oxamide dioxime.** M. Kuraš and J. Mollin (Chem. Ústav, Vysoká Škola Pedagogická, Olomouc, Czechoslovakia). *Chem. Listy*, 1958, **52** (2), 344–346.—Aq. ethanolic soln. of oxamide dioxime (**I**) yield two waves in the acid range of Britton-Robinson buffer soln., the first of which is of diffusion character and is suitable for analytical purposes ($E_{\frac{1}{2}} = -0.434$ V in aq. soln., $E_{\frac{1}{2}} = -0.692$ V in aq. ethanolic soln.). The amperometric titration of Ni^{2+} can be carried out at -1.14 to -1.20 V, in aq. NH_3 soln., with the use of 0·05 *M* soln. of **I**. *Procedure*—Dilute 1 to 20 ml of the $NiCl_2$ soln. (0·3 to 0·6 g of Ni^{2+} in 1 litre) with a twofold vol. of 0·2 *M* NH_4Cl, 0·15 *M* aq. NH_3 (40 ml) and gelatin soln. (1%) (1 ml). Titrate with 0·05 *M* **I** with stirring under nitrogen. Dropping mercury and satd. calomel electrodes were used. Interference is caused by Cu^{2+}, Fe^{2+}, Fe^{3+}, Co^{2+} and Cr^{3+} owing to reaction with **I**; Mn^{2+}, Al^{3+}, Zn^{2+}, Pb^{2+} and Ag^+ can be masked with citric or tartaric acid. Cyanides must be absent. J. ZÝKA

3757. **Separation of fission products by the distillation method. III. Separation of carrier-free ruthenium with ammonium persulphate as oxidising agent.** Tomihisa Kambara (Fac. of Educ., Shizuoka Univ., Oiwa-cho). *Japan Analyst*, 1958, **7** (2), 89–93.—When distilled from an HNO_3 soln. (sp. gr. 1·38, 8 ml per 45 ml) containing $AgNO_3$ (1 *M*, 1 ml per 45 ml) and $(NH_4)_2S_2O_8$ (10 g), Ru in fission products is collected almost quant. ($\simeq 95\%$) in the distillate within 9 min. in a current of air. Only a small amount of SO_4^{2-} (< 0.5 mg) is distilled over. Silver peroxide can also be used as oxidising agent. The decontamination factor is $\simeq 10^5$. K. SAITO

3758. **Amperometric determination of palladium with β-furfuraldoxime.** M. B. Bardin and N. D. Meleka. *Uch. Zap. Kishinevsk. Univ.*, 1957, **27**, 87–91; *Ref. Zhur., Khim.*, 1958, Abstr. No. 7614.—Replacement of the dropping electrode with a solid electrode permits the polarographic determination of the platinum metals from soln. of their usual salts, without the use of complex formation. Satisfactory results are obtained in the amperometric determination of Pd with β-furfuraldoxime on the rotating platinum electrode. In a basal soln. of 0·1 *N* $NaNO_3$, β-furfuraldoxime is not reduced on this electrode. In a measuring flask of 50-ml capacity, place an aliquot of a standard palladium soln., and 0·5 ml of *N* HCl, and make up to the mark with the background soln. Transfer 10 ml of the soln. obtained to the polarographic cell and pass a current of N. Then add the reagent from a micro-burette, at a potential of 0·9 to 1·0 V. The current is noted 40 sec. after the addition of the reagent. The titration curves are L-shaped. The end-point is clear; good reproducibility of results is observed if the acidity of the soln. is $\geq$ 0·1 *N* in HCl. The concn. of Pd in the soln. is directly proportional to the volume of reagent used. At a concn. of 10^{-3} to 10^{-4} *M* Pd the error is $\pm$ 3%. C. D. KOPKIN

3759. **Direct determination of mineral matter in coal.** M. Bishop and D. L. Ward (National Coal Board, London, S.E.11). *Fuel, Lond.*, 1958, **37** (2), 191–200.—The Rademacher and Mohrhauer method, based on extraction with conc. HCl and HF (*cf. Anal. Abstr.*, 1956, **3**, 124), has been investigated. Good duplication of results was possible, and a standard deviation of $\pm$ 0·2% between laboratories was obtained. The only modification recommended is the use of poly(vinyl chloride) for the filtration unit. P. M. SORGO

3760. **Spectrographic analysis of ores by introducing the powder into the arc in a stream of air.** A. K. Rusanov and V. G. Khitrov (Inst. of Appl. Mineral. USSR, Moscow). *Spectrochim. Acta*, 1958, **10** (4), 404–418.—The powder, carried in a stream of air, is introduced evenly into the discharge zone of a horizontal a.c. arc (*cf. Anal. Abstr.*, 1956, **3**, 1561). This procedure prevents reactions that normally occur on the electrodes, prevents the fractional evaporation of different elements and ensures uniform conditions of excitation. The excitation conditions are determined mainly by the ionisation potentials of the elements in the sample and depend very little on the composition of the electrodes. The technique gives a significant increase in sensitivity and reproducibility and reduces the time required for analysis. Direct photo-electric measurement of line intensities is possible. A. R. ROGERS

3761. **Theory of the testing of alloys by surface treatment.** A. N. Tananaeva. *Trudy Ural'sk. Politekh. Inst.*, 1957, (69), 143–147; *Ref. Zhur., Khim.*, 1958, Abstr. No. 21,083.—The surface dissolution of steels is reviewed on the basis of the theory of electrochemical processes and metallographic considerations. In the dissolution of alloys with a homogeneous structure the soln. is the same as that of the original alloy. In alloys with a heterogeneous structure, the dissolved and undissolved portions (in steels the ferrous base and the carbides) are analysed. The solution mechanism for low-, medium- and high-alloyed steels is the same. C. D. KOPKIN

See also Abstracts—**3588**, Use of phthalein complexone in determinations of Cd and Mn. **3589**, Styrylcupferron: reagent for Cu, Fe and Al. **3590**, Extraction of U with methyl*cyclo*hexanone. **3592**, Charcoal-block reactions in qualitative analysis. **3593**, Metallochromic indicators. **3595**, Glycine-naphthol violet as chelatometric indicator. **3765**, Determination of O in coal. **3822**, Determination of Hg. **3823**, **3824**, Spectrophotometric determination of Fe. **3921**, Determination of Ga. **3922**, Determination of Ge.

3.—ORGANIC ANALYSIS

3762. **Chloraminometry—a new method of organic analysis.** B. N. Afanas'ev. *Trudy Sverdl. S.-Kh. Inst.*, 1957, **1**, 361–368; *Ref. Zhur., Khim.*, 1958, Abstr. No. 11,039.—Standard soln. of chloramine T, which are completely stable on long storage in dark-glass flasks and on heating to 60°, are used for the determination of various organic substances. A method is proposed for the direct titration with chloramine T in the presence of indigo carmine as indicator, in an acid medium, of polyhydric alcohols, citric and oxalic acids and thiourea, and, in an

alkaline medium, of aldehydes, urea and glucose. By reason of their low oxidisability, ketones are determined indirectly by formation of their bisulphite addition compounds, with titration of excess of bisulphite with chloramine T. An indirect chloraminometric method is evolved for the determination of phenylhydrazine, and of carbon disulphide, based on its conversion in an alcoholic alkaline medium into K ethylxanthate, which is titrated directly with chloramine T.

C. D. KOPKIN

3763. **Aspects of organic analysis. VI. The micro-determination of active hydrogen in highly insoluble substances.** G. W. Perold and J. M. Snyman (S. A. Iron and Steel Ind. Corp. Ltd., P.O. Box 450, Pretoria, S. Africa). *Mikrochim. Acta*, 1958, (2), 225–227 (in English).—Kairoline(1:2:3:4-tetrahydro-1-methylquinoline) is proposed as a powerful solvent in the Zerewitnoff micro-determination of active hydrogen in highly insol. substituted aromatic amides and terpenoid hydroxy acids. Detailed modifications to the apparatus and procedure of Pregl for the Zerewitnoff determination are described. D. F. PHILLIPS

3764. **New vacuum method for the determination of carbon and hydrogen in organic substances.** P. N. Fedoseev and L. S. Ignatenko. *Izv. Akad. Nauk, Turkm.SSR*, 1957, (3), 24–30; *Ref. Zhur., Khim.*, 1958, Abstr. No. 978.—The method described is based on the decomposition of the sample in an evacuated tube by heating with CuO, with subsequent ignition of the pyrolysis products in a current of O or air. By this method, volatile, readily decomposable and N-containing substances are easily, rapidly and reliably analysed; the error in determining C is ± 0·15 to 0·30%, and in determining H is ± 0·10 to 0·20%. Eight to ten ignitions may be carried out in one working day.

C. D. KOPKIN

3765. **Direct determination of oxygen in organic substances and in coal.** C. Siniramed and C. Manci (Exptl. Sta. Combustibles, Milan). *Riv. Combustibili*, 1957, **11**, 366–379.—The method and apparatus of Spooner and Georgiadis were modified to improve accuracy and precision. The apparatus consisted of (*i*) a purification train for carrier N, (*ii*) a combustion tube packed with charcoal, and (*iii*) an absorption train for product gases, including a catalytic CO to CO_2 converter. Blanks were ≃ 0·15% of O. Successive determinations of O in benzoic acid (theoretical value 26·20%) were 26·20 ± 0·13%, in phenylacetamide (11·80%) 11·90 ± 0·60%, in cetyl alcohol (6·60%) 6·52 ± 0·02%, and in chrysene (0%) 0·10 ± 0·06%. Calcium carbonate (43·8% of CO_2) gave 43·78 ± 0·02%. Of the commercial products tested, Venezuela coal-tar gave 3·30% of O, aromatic lubricating extract 0·5%, synthetic hydrocarbon polymers 0·07 to 0·17%, lignite 17·85%, and Pocabontas coal 1·46%. CHEM. ABSTR.

3766. **New variant of the Dumas method of determining nitrogen.** V. A. Klimova and I. F. Dubinina (N. D. Zelinskiĭ Inst. of Org. Chem., Acad. Sci., USSR). *Izv. Akad. Nauk, SSSR, Otd. Khim. Nauk*, 1958, (2), 129–132.—The sample is placed in a small silica tube (90 mm long and 5 mm in internal diameter), closed at one end, and mixed with a mixture of CuO and NiO (100:1). The tube is then rather more than half filled with the same mixture and the remaining space is filled with reduced copper powder. The tube with its open end facing the connection to the supply of CO_2 (from a Kipp's apparatus) is then placed in the middle of a 40-cm combustion tube, empty except for a 6-cm length of Hopcalite (to oxidise CO) maintained at 150° and situated towards the outlet end of the combustion tube. The nitrometer is connected and the small silica tube is heated at 750° to 800° so that the copper and some of the oxide mixture attains the temp. first. The method is accurate to ± 0·2% of the sample, even when the ordinary Dumas method is very inaccurate, *e.g.* for decahydro-4-hydroxyquinoline hydrochloride and $AgNO_3$. G. S. SMITH

3767. **The Kjeldahl - Ronchése method for the rapid determination of organic nitrogen.** R. F. Gómez Vigide (Inst. Alonso Barba, Fac. de Farm., Santiago). *Inf. Quim. Anal.*, 1958, **12** (1), 9–12.—Low results by the Ronchése method for the determination of NH_3 after digestion of the sample with H_2SO_4 are due to the presence of secondary and tertiary amines. These do not interfere in the normal distillation method since they behave as NH_3 for titration purposes, but they do not react completely with formaldehyde. The use of Hg as catalyst and a longer digestion time ensures conversion of all nitrogen into NH_4^+, and the Ronchése method gives results within ± 1% of theory for several pharmaceutical chemicals, on both the semi-micro and the micro scale. *Procedure* (*micro-method*)—The sample (containing 2 to 5 mg of N) is digested with 2 g of K_2SO_4, 0·2 g of HgO and 4 ml of conc. H_2SO_4 until the mixture is colourless and then for a further 10 min. The mixture is diluted with 20 ml of H_2O and 4 ml of 60% NaBr soln. (to prevent interference by Hg) and 2 drops of methyl red are added. Most of the excess of acid is neutralised by addition of 40% NaOH soln., the mixture is boiled to remove CO_2, and finally neutralised to a yellow end-point with 0·02 *N* NaOH. Formaldehyde soln. (previously neutralised to phenolphthalein) (18%) (8 to 10 ml) is now added and the acid liberated is titrated to a phenolphthalein end-point. A blank determination is carried out at the same time. G. H. FOXLEY

3768. **Determination of halogens (chlorine, bromine, iodine) and sulphur on a single sample by a modification of the micro-method described by W. Schöniger.** M. Boëtius, G. Gutbier and H. Reith (Tech. Hochschule, Dresden). *Mikrochim. Acta*, 1958, (3), 321–324 (in German).—The simultaneous determination of sulphur and halogens by the Schöniger method has previously been possible only if the substance does not contain N. A procedure is described which enables the method to be applied to all organic compounds. An Erlenmeyer flask is charged with 5 ml of 0·5 *N* NaOH and 4 to 5 drops of H_2O_2 (30%, H_2SO_4-free). The substance is then burnt on platinum gauze in the usual way and the flask shaken for 10 min. to dissolve the combustion gases. After rinsing the gauze with twice-distilled water, the soln. is boiled for 2 min. to remove H_2O_2, treated with 3 ml of acetic acid (210 ml of glacial acetic acid with 300 ml of water) and re-heated to expel CO_2. Methanol (10 ml) and 3 drops of alizarin red S (0·1% aq. soln.) are added to the cooled soln., which becomes yellow. The soln. is then titrated with 0·02 *N* $Ba(NO_3)_2$ until the indicator becomes pink, and all the SO_4^{2-} have been pptd. as $BaSO_4$. Finally, the halogen is determined by the Volhard method. Satisfactory results are obtained for the determination of chloride but, for the titration of bromide,

results are 1% higher than theoretical. Correct figures are obtained by using an empirical factor of 1·569 (for 0·02 *N* $AgNO_3$) instead of the theoretical 1·589. Experiment has confirmed that, for the iodide determination, when iodine dissolves as both iodate and iodide, errors are not introduced by the pptn. of barium iodate or by the reduction of iodide to iodine by iodate in the presence of acid. Iodine is determined after titration with $Ba(NO_3)_2$ soln. by adding 15 ml of bromoacetic acid soln., the excess of which is reduced to bromine with formic acid and titrated with 0·02 *N* $Na_2S_2O_3$ after addition of KI and 6 ml of 2 *N* H_2SO_4. The limits of error of the procedure are ± 0·3%. S. M. MARSH

3769. **Semi-micro gravimetric determination of sulphur in organic substances by combustion.** A. Étienne and J. Léger (Lab. de Chim. Org., l'École Pratique des Hautes Études, Coll. de France). *Chim. Anal.*, 1958, **40** (2), 43–46.—Burn the sample in a rapid stream of O by the empty tube technique and trap the sulphur oxides as Ag_2SO_4 on a coil of silver wire maintained at 550°. The wire, of 0·4 mm diameter, is coiled into a cylinder (5 cm × 1 cm) and is supported in a cylinder of 80-mesh platinum gauze. A coil weighing 10 g will trap quant. about 16 mg of S. If the sample does not contain halogens, measure the gain in wt. of the coil during the combustion. If the sample contains halogens, extract the Ag_2SO_4 from the coil by boiling with H_2O (100 ml) and measure the loss of wt. of the coil on extraction. The percentage of S in the sample determined by this method is correct within ± 0·1. A. R. ROGERS

3770. **Micro-analytical determination of acetyl groups.** A. S. Inglis (Defence Standards Lab., Dept. of Supply, Maribyrnong, Victoria, Australia). *Mikrochim. Acta*, 1958, (2), 228–235 (in English).—It has been confirmed that the apparatus due to Schöniger *et al.* (*Mikrochim. Acta*, 1954, 96) gives a simple, rapid and quantitative distillation of acetic acid. It is shown that the *N* methanolic NaOH reagent is not as effective as has previously been thought and that 5 *N* NaOH in methanol (33%) is more suitable for acetyl determinations. For acetylated carbohydrate analysis, contrary to earlier reports, the alkalimetric titration of acetic acid was found to be superior to the iodimetric titration (Friedrich and Sternberg, *Biochem. Z.*, 1936, **285**, 20) provided that the correct blank value is used for the de-acetylated parent substance. D. F. PHILLIPS

3771. **Determination of organic acids and carbonyl compounds.** Stanley Satoshi Yamamura (Iowa State Coll., Ames, U.S.A.). *Dissert. Abstr.*, 1957, **17** (12), 2787–2788.—By using tetra-alkylammonium hydroxides in benzene - methanol as titrants, a wide variety of organic acids can be titrated in non-aqueous solvents; the reaction is followed potentiometrically. Five classes of these, and several mineral acids, have been examined and the effect of various substituents studied, the number and position of the groups in relation to the acidic group being varied. Numerous phenols, carboxylic acids, enols, imides and sulphonamides were also titrated potentiometrically in acetone and methyl cyanide. A differentiating titration is possible if the p.d. between the beginnings of the inflections of the stronger and weaker acids is > 100 mV. Aldehydes and ketones can be analysed by using a 1:1 mixture of hydroxylamine and hydroxylamine hydrochloride in abs. *iso*propyl alcohol as the reagent; when the reaction is complete, the excess of reagent is titrated potentiometrically with alcoholic HCl. With carbonyl compounds, good precision is attainable, there being a sharp break at the equivalence point. It is possible to analyse samples containing equimolar amounts of carboxylic acids and esters; amines and high concn. of these acids produce low results. S. C. I. ABSTR.

3772. **Preliminary test for nitrogen- and oxygen-containing groups in organic compounds.** F. Feigl and J. R. Amaral (Min. of Agric., Rio de Janeiro, Brazil). *Mikrochim. Acta*, 1958, (3), 337–341 (in English).—Oximes, hydroxamic acids, amine oxides, nitro, nitroso and azoxy compounds evolve HNO_2 when heated to dryness in a test-tube for ≃ 1 or 2 min. The acid vapour can be readily detected by a filter-paper moistened with Griess reagent (equal vol. of a 1% soln. of sulphanilic acid and a 0·1% soln. of 1-naphthylamine, both in 30% acetic acid). Limits of detection are from 0·2 to 50 μg (11 compounds listed); 30 compounds that give positive results with samples of < 1 mg are also listed. The test is valid for organometallic compounds *e.g.*, Cu benzoin oxime, Ca picrolonate. W. J. BAKER

3773. **The intensities of the infra-red absorption bands of *n*-paraffin hydrocarbons.** R. N. Jones (Div. Pure Chem., Nat. Res. Counc., Ottawa, Canada). *Spectrochim. Acta*, 1957, **9** (3), 235–251.—The mol. extinction coefficients of the principal absorption bands in the spectra of the homologous series of *n*-paraffin hydrocarbons from hexane to hexatriacontane have been measured. The values of $\epsilon_{max}^{(a)}$, when plotted against the number of methylene groups in the chain, yield linear graphs with positive slopes. An analysis of the band envelope in the region 1490 to 1440 cm^{-1} indicates the presence of a third peak near 1460 cm^{-1} in addition to the well-established methylene and methyl bands at 1467 and 1457 cm^{-1}. N. E.

3774. **Fractometric determination of methane.** L. Coppens, J. Bricteux and J. Venter. *Bull. Tech. Houille, Liége*, 1958, (13), 426–446.—Application of gas-chromatographic adsorption methods to the quant. determination of methane in methane - air mixtures was investigated. By the use of Linde 5A molecular sieves as adsorbent, methane can be determined extremely rapidly. Results are accurate and free from interference by other components. FUEL ABSTR.

3775. **Separation and determination of the higher acetylenes produced by the electric arc cracking of methane gas by gas - liquid partition chromatography.** H. Gunesch and R. Stadtmüller. *Rev. Chim., Bucharest*, 1958, **9** (1), 35–38.—The method described is claimed to be simple to operate and to detect the components of a gaseous mixture even when some are in extremely small concn. The operation takes only a few minutes and, as the action is completely reversible, the chromatographic column can be re-used many times. Standardisation of the apparatus with known mixtures is not necessary, and continuous automatic analyses are possible by the use of a thermal conductivity cell as a detecting instrument. H. SHER

3776. **Determination of small amounts of methanol in ethanol and fruit brandies by the method of Denigès.** G. Rankov, A. Popov and A. Iovchev

(Acad. Bulgare Sci., Sofia). *Compt. Rend. Acad. Bulgare Sci.*, 1955, **8** (2), 53–56 (in German).—The method of Denigès is used with some slight modifications. N. E.

3777. **Determination of methanol in the presence of ethanol, water and other volatile substances.** G. Rankov, A. Popov and A. Iovchev (Acad. Bulgare Sci., Sofia). *Compt. Rend. Acad. Bulgare Sci.*, 1958, **11** (1), 49–52 (in German).—A method is described for the preparation of ethanol (**I**) that is free from methanol (**II**). To determine **II** in aq. soln. in the presence of **I**, acetaldehyde, esters, acids and higher alcohols, dilute the sample with H_2O until the concn. of **I** is 10% and add a 10% aq. soln. of **II**-free **I** until the concn. of other volatile substances is less than one-hundredth that of **I**; complete the determination by the method of Rankov *et al.* (*Anal. Abstr.*, 1958, **5**, 3776), with Schiff's reagent. Alternatively, work at a concn. of 5% of **I** and determine **II** by the use of chromotropic acid reagent. A. R. Rogers

3778. **Determination of small quantities of glucose in the presence of large quantities of lactose.** K. I. Evstratova (Leningrad Chem. Pharm. Inst.). *Biokhimiya*, 1958, **23** (2), 181–184.—Into a 150-ml flask measure Cu acetate soln. (48 g per litre, 20 ml), add 10% lactic acid soln. (3·5 ml) and 20% Na acetate soln. (10 ml), followed by 20 ml of the sugar soln. (containing up to 1·75% of lactose). Boil for exactly 5 min. from the time of appearance of the first bubbles. Rapidly cool under the tap and determine the cuprous oxide by Bertrand's method. The amount of glucose is found from a calibration table (given). By this method glucose in concn. of 0·01 to 0·5% can be determined in the presence of a concn. of 1·75% of lactose. C. D. Kopkin

3779. **Analysis of sodium formaldehyde-bisulphite and sodium formaldehydesulphoxylate (rongalite).** L. Maros (Inst. for Inorg. and Anal. Chem., Loránd Eötvös Sci. Univ., Budapest). *Magyar Kém. Foly.*, 1958, **64** (2), 41–44.—Sodium formaldehydesulphoxylate (**I**) is oxidised in a slightly acid soln. by iodine, but sodium formaldehyde-bisulphite (**II**) is not. In alkaline medium KCN decomposes **II** into SO_3^{2-} and the salt of glycollic acid; the SO_3^{2-} can be determined iodimetrically. *Procedure*—For the determination of **II**, dissolve the sodium salt (0·7 to 0·8 g) in H_2O (100 ml). Pipette an aliquot (10 ml) into a 200-ml flask and wash the sides of the flask with a little H_2O. Add pentane (5 ml, to exclude oxygen), NaOH soln. (10%) (2 ml) and KCN (0·2 to 0·3 g). Stir and, after 2 to 3 min., dilute with H_2O to 120 ml. Add methyl orange (1 drop), then H_2SO_4 (50%) dropwise until the soln. becomes acid, then 2 to 3 drops in excess. Add KI (0·2 g) and starch, and titrate with 0·1 *N* iodine to a violet end-point. If > 10 ml of iodine is used, no pentane is needed as the error caused by air is > 0·5%. For the simultaneous determination of **I** and **II**, dissolve a sample (0·4 g) of **I** containing **II** in H_2O (100 ml). Transfer 10 ml to a 100-ml flask, dilute to 40 ml with H_2O; add formaldehyde soln. (40%) (0·5 ml), Na acetate soln. (20%) (1 ml) and acetic acid soln. (10%) (1 ml). Titrate with 0·1 *N* iodine in the presence of starch. Another aliquot is determined as described earlier; this gives the concn. of **I** plus **II**; the concn. of **II** is obtained by subtraction. Formaldehyde does not interfere in any of these titrations. A. G. Peto

3780. **Estimation of di- and tri-chloroacetaldehydes in the presence of each other.** O. P. Malhotra and V. D. Anand (Hindu Univ., Benares, India). *Z. anal. Chem.*, 1958, **160** (1), 10–14 (in English).—Three methods are presented. In the first, the sum of the aldehydes is determined in two ways. A sample is heated with 0·5 *N* KOH and the excess of KOH is determined. Another sample is shaken with KOH soln. and iodine, and the excess of iodine is determined. The separate amounts of the two aldehydes can be obtained by solution of the simultaneous equations for the two determinations. This method yields results within 4·5% of the true value (from four determinations) with neutral aq. soln. In the second method, trichloroacetaldehyde is determined colorimetrically by heating a sample with NaOH soln., pyridine and acetone, and matching the colour of the pyridine layer against glass standards. The sum of the aldehydes is determined iodimetrically. Results are within 1·5% for dichloroacetaldehyde and within 1·2% for trichloroacetaldehyde (from five determinations) in aq. soln. In the third method, the trichloroacetaldehyde is determined colorimetrically, as described for the second method, then another sample is hydrolysed with KOH, neutralised with H_2SO_4, and the Cl^- determined by titration with $AgNO_3$ soln. This method yields results within 3·2% for dichloroacetaldehyde (from four determinations) in aq. or aq. ethanolic soln. in the absence of free acid. The choice of method depends on whether the soln. contains free acid or ethanol. G. Burger

3781. **Separation and determination of mono-, di- and tri-chloroacetic acids with an ion-exchange resin.** Wataru Funasaka, Tsuguo Kojima, Michihiro Ishibashi and Hajime Kita (Fac. of Engng, Kyoto Univ., Sakyo-ku). *Japan Analyst*, 1958, **7** (2), 69–73.—Mono-(**I**), di-(**II**) and tri-chloroacetic acid (**III**) (< 2 milli-equiv. each) are eluted from a column (diam. 1·2 cm, height 20 cm) of Amberlite IRA-410 (NO_3^-) (100 to 150 mesh) with, respectively, 0·05 *M*, 0·1 *M* and *M* $NaNO_3$ (250 ml each). Each eluate is boiled with NaOH (30 g per 100 ml of the eluate) for 1 hr., then acidified with conc. HNO_3, neutralised with aq. NH_3 soln. (sp. gr. 0·88) (to precipitate SiO_2), treated with $Ca(NO_3)_2$ soln. (10%, 5 ml), filtered and titrated by the Volhard method. This method is applicable to the analysis of industrial **I**. The use of a resin in the carbonate form is less satisfactory. K. Saito

3782. **Oxidimetric determination of tartaric acid and tartrates.** E. Krejzová, V. Simon and J. Zýka (Dept. Anal. Chem., Charles' Univ., Prague). *Českosl. Farm.*, 1958, **7** (2), 82–83.—Indirect oxidimetric determinations of tartaric acid and tartrates with $K_2Cr_2O_7$ or ceric salts were studied, and the dichromate was found to be more suitable as a reagent. The excess of dichromate is back-titrated with 0·1 *N* quinol, with potentiometric control. The procedures are given in detail. J. Volke

3783. **Potentiometric determination of vinyl cyanide by means of sodium sulphite.** A. P. Terent'ev, S. I. Obtemperanskaya and M. M. Buzlanova (M. V. Lomonosov Moscow State Univ.). *Zavod. Lab.*, 1958, **24** (2), 157.—Reaction of vinyl cyanide with Na_2SO_3 yields NaOH, which can be titrated potentiometrically even in coloured soln. The sample (1 g) of dried (over $CaCl_2$) and distilled vinyl cyanide in a sealed ampoule is placed in a 250-ml calibrated flask together with 25 ml of

dioxan (distilled over Na) and 25 ml of 0·5 *N* Na_2SO_3. The ampoule is broken, the soln. is made up to the mark with 0·5 *N* Na_2SO_3 and titrated potentiometrically after 5 min. with 0·1 *N* HCl, a 25-ml aliquot mixed with 10 ml of water being used. No interference is caused by the presence of 2-cyanoethanol. G. S. SMITH

3784. **Adaptation to plant control of the method of analysing mixtures of ammonia and methylamines.** J. Issoire. *Chim. et Ind.*, 1958, **79** (2), 163–172.—In the determination of monomethylamine (**I**), dimethylamine (**II**) and trimethylamine (**III**) in the presence of NH_3, the aq. soln. (50 ml) of the ammonium salts and amines is allowed to react (without stirring) with aq. 33% $NaNO_2$ soln. (40 ml) and glacial acetic acid (10 ml) at 30° for 1 hr., whereby NH_3 and **I** are converted into N, **II** into its nitroso derivative, whilst **III** is unchanged. After removal of nitrous vapours, the mixture is made strongly alkaline and **III** is removed by distillation and determined acidimetrically (the abs. error is 0·1 to 0·2%); **II**, which distils over simultaneously as the nitroso compound, can be determined directly by polarography in HCl soln.; $E_{\frac{1}{2}}$ is $\simeq$ 0·82 V, and a calibration curve for pure dimethylamine hydrochloride is used; the error is 1 to 2%. The NH_3 must be determined on a separate sample—(*i*) for concn. $\leqslant$ 80% (w/v) the amines are passed slowly into a well-cooled soln. of HCl ($\simeq$ 2 *N*) in a mixture (1 + 3, by vol.) of methanol and $CHCl_3$; when the neutral point is reached the NH_4Cl ($\equiv$ NH_3 present) is collected and weighed; $\geqslant$ 20% (w/v) of **I** should be present; (*ii*) for concn. < 2% (w/v) the Nessler colorimetric method is used, but the error is 5 to 10%, depending on the amine content; (*iii*) average concn. of NH_3 can be determined by pptn. with sodium cobaltinitrite at 5°. The percentage of **I** is finally obtained by difference. One analysis takes 3 to 4 hr.; the total content of amines should be determined initially by the distillation method (the error is 0·2%); the sample soln. taken should be approx. *N* in amine content. Precautions, sources of error and necessary corrections, *e.g.*, for NH_3 formation during nitrosation of **I**, are discussed.
W. J. BAKER

3785. **Application of nitrometry. XVI. Quantitative determination of secondary amines.** Makoto Yokoo (Res. Lab., Takeda Pharm. Ind. Ltd.). *Chem. Pharm. Bull., Japan*, 1958, **6** (1), 64–66 (in English).—Diphenylamine, proline, kainic acid, and the hydrochlorides of dimethylamine, diethylamine and dibenzylamine can be determined by this method with an accuracy of ± 1%. Primary and tertiary amines do not interfere. *Procedure*—Dissolve the sample (10^{-4} mole) in H_2O (1 ml) and glacial acetic acid (0·5 ml), add 60% aq. KNO_2 soln. (0·5 ml) and allow to stand for 30 min. at 30°. Cool, and at the same time add a slight excess of 20% aq. sulphamic acid (1 or 1·5 ml), followed by powdered zinc (2 g) added with shaking. Add conc. HCl (4 ml) portion-wise during 30 min. with constant shaking, then add Devarda's alloy powder (0·5 or 1 g) and allow to stand for 30 min. Filter through sintered glass, dilute the filtrate to 50 ml with H_2O, and measure the vol. of N liberated from a 1-ml aliquot by the action of alkaline $K_3Fe(CN)_6$.
A. R. ROGERS

3786. **Turbidimetric and spectrophotometric titration of ethylenediaminetetra-acetic acid with lead nitrate, with chromate ions as indicator.** Soichiro Musha, Makoto Munemori and Kinya Ogawa (Fac. of Engng, Univ. of Osaka Prefecture, Sakai). *Japan Analyst*, 1958, **7** (1), 49–50.—By the use of an electrophotometer the end-point of the titration of EDTA (several tenths of a mg) with Pb^{2+} can be indicated with K_2CrO_4 soln. (5%), either photometrically (610 mμ) or turbidimetrically without filter. The error is < 1%, without a protective colloid. K. SAITO

3787. **Analytical separation and detection of flavianic acid and rubeanic acid [dithio-oxamide].** K. Suchý (Balneological Inst., Charles' Univ., Prague). *Magyar Kém. Foly.*, 1958, **64** (2), 46–48.—The polarographic behaviour of flavianic acid (**I**) and dithio-oxamide (**II**) was examined. For the electrolysis, a Kalousek vessel was used, and a normal calomel electrode. To *N* H_2SO_4, containing aq. fuchsine (0·02%), a 0·01 *N* soln. of **I** or **II** in pyridine was added; the waves were suitable for polarographic determination. As their $E_{\frac{1}{2}}$ values differ, **I** and **II** can be determined simultaneously. On standing, the soln. of both **I** and **II** decompose by a reversible unimolecular mechanism; thus they cannot be used for the polarographic titration of metals such as Co, as has been suggested.
A. G. PETO

3788. **Detection of carbonyl compounds in benzene.** D. A. Forss and E. A. Dunstone (Dairy Res. Section, C.S.I.R.O., Melbourne, Australia). *Chem. & Ind.*, 1958, (5), 127–128.—To a chromatographic column (20 cm × 0·5 cm) containing 3 ml of the benzene sample, and packed with prepared activated alumina to a height of 10 cm, is added 0·2 mg of 2:4-dinitrophenylhydrazine dissolved in 1 ml of the sample. When this has been taken up, a further 1 ml of the benzene is added, and the column is developed with 3 ml of the benzene. If the sample is carbonyl-free, the deep-red hydrazine zone remains at the top, or moves only very slowly; if a trace of carbonyl is present, the resulting yellow hydrazone is leached from the red band. Figures are given for the behaviour of 10 carbonyl compounds dissolved in purified benzene. As little as 0·00025% of ethyl methyl ketone can be detected as a yellow band, 10 mm wide, below the red.
R. E. ESSERY

3789. **Quantitative determination of 1:2-epoxides.** M. Mousseron, J. Jullien and A. Peyron. *Parfum. Cosmét. Savons*, 1958, **13** (1), 3.—The mechanism of epoxide reactions and their applicability as a basis for quantitative analysis is discussed. Under anhydrous conditions the epoxides are determined acidimetrically. It is shown that the method may also be applied when there is a possibility of hydration. *Procedure*—To 10 ml of satd. KSCN soln. (210 g per 100 ml of water) add the epoxide soln. (5 to 10 ml, 0·1 to 0·2 *N* epoxide in ethanol - H_2O or dioxan - H_2O soln., equal parts). Mix the soln. thoroughly, add *N* HCl (5 ml) then bromophenol blue (1% in ethanol, 5 drops). Stir well and titrate with 0·2 *N* NaOH. The difference in volume of 0·2 *N* NaOH used by a blank and the epoxide soln. corresponds to the epoxide present. Limitations of the assay when applied to several compounds are discussed. H. B. HEATH

3790. **Use of the skeletal nickel catalyst in the analysis of a series of organic compounds.** N. S. Goryacheva. *Med. Prom., SSSR*, 1957, (3), 32–35; *Ref. Zhur., Khim.*, 1958, Abstr. No. 7695.—The skeletal nickel catalyst is used for the quant.

reduction of phenylhydrazine and its derivatives substituted in the benzene ring, acid hydrazides, semicarbazide derivatives and nitrobenzene reduction intermediates. Boil the sample (0·1 g) for 1 hr. with 0·5 *N* ethanolic KOH (25 ml) and 2 or 3 g of the active catalyst. After evaporation of the ethanol, titrate the resulting amine with $NaNO_2$ soln. In the analysis of iodophenylhydrazine, carry out the titration after steam-distillation of the aniline formed; for diazoaminobenzene carry out the titration at 0°. In the determination of isoniazid, use 5 g of catalyst; the reduction is carried out in a special apparatus (illustrated). Steam-distil the NH_3 formed into a receiver containing a standard acid soln., and titrate the excess of acid with alkali to methyl red.

C. D. KOPKIN

3791. **Analysis of alkylbenzenesulphonates.** E. W. Blank (Colgate-Palmolive Co., Jersey City, N.Y., U.S.A.). *Soap, N.Y.*, 1958, **34** (1), 41–47, 107.—A critical survey of methods for analysing sodium dodecyl- (or pentadecyl-)benzenesulphonate is made. Characterisation of the alkylbenzenesulphonate for subsequent use in standardising the cationic reagent is dealt with, sampling of slurries and powders is described, and full analytical details of apparatus, reagents, procedure and calculations are given for the determination of alcohol-soluble material, determination of active ingredient (Na alkylbenzenesulphonate) by cationic titration, and determination of neutral oil and water. G. HELMS

3792. **Colorimetric method for determining low concentrations of mercaptans [thiols].** G. L. Ellman (Dow Chemical Co., Midland, Mich., U.S.A.). *Arch. Biochem. Biophys.*, 1958, **74** (2), 443–450.—The method is based on the quant. reaction of many thiols with di-(*p*-nitrophenyl) disulphide (**I**) to liberate *p*-nitrobenzenethiol, which can be determined in aq. soln. at pH 8·0 from the extinction at 412 mμ. *Reagent*—Recrystallise commercial **I** from glacial acetic acid to m.p. 183° to 184° and dissolve 30·8 mg in 100 ml of acetone. *Procedure*—To 4·0 ml of test soln., which should be neutral and practically salt-free, add 1·0 ml of 0·1 *M* phosphate buffer (pH 8·0), 4·0 ml of acetone and 1·0 ml of reagent. Determine the extinction at 412 mμ. Simple thiols produce a stable colour immediately; cysteine and related mercapto compounds require 60 to 90 min. Calibrate with *p*-nitrobenzenethiol purified by dissolution in 5% aq. Na_2CO_3 and filtration into *N* HCl. Recrystallise from glacial acetic acid to m.p. 77° to 79°. The method will determine 2 to 4 × 10^{-6} mole, or about 1 μg of cysteine. The specificity, interferences and the kinetics of the reaction are discussed.

W. H. C. SHAW

3793. **Bromatometric determination of some 2-naphtholsulphonic acids (Schäffer's acid, R-acid, G-acid) [2-naphthol-6-sulphonic, 2-naphthol-3:6-disulphonic, 2-naphthol-6:8-disulphonic acids].** J. Laszlovszky (Inst. for Inorg. and Anal. Chem., Loránd Eötvös Sci. Univ., Budapest). *Magyar Kém. Foly.*, 1958, **64** (1), 5–9.—Salts of Schäffer's acid (**I**) and R-acid (**II**) can be determined by direct titration with $KBrO_3$ or by indirect bromination; the time of the bromination of **I** must not exceed 1 or 2 min. *Procedure*—For the direct titration of **I** or **II**, dissolve ≃ 1 milli-equiv. of the salts in water (30 to 40 ml) with gentle warming. Add KBr (1 g) and acidify with acid (5 ml), 5 *N* H_2SO_4 for **I** and 5 *N* HCl for **II**. Warm the soln. to between 40° and 50°, add 0·2% *p*-ethoxychrysoidine (1 drop) and titrate slowly with 0·1 *N* $KBrO_3$ until the soln. becomes lemon yellow. For the indirect titration of **I** or **II**, dissolve ≃ 1 milli-equiv. in water (30 to 40 ml), with warming, in a flask with a ground glass stopper. Cool the soln., add KBr (1 g) and a ≃ 100% excess of 0·1 *N* $KBrO_3$ with an accuracy of 0·01 ml. Then acidify the soln. quickly with 5 *N* HCl (5 ml) and stopper the flask immediately; shake it and add, with a rapid movement, KI (1 g). After 5 min., titrate the iodine with 0·1 *N* $Na_2S_2O_3$. The bromination of G-acid (**III**) by either of these two methods is very slow and so the determination is not possible; but, in the presence of not more than half of its wt. of **III**, **II** can be determined by the indirect method if the time of bromination is kept very brief.

A. G. PETO

3794. **Mass spectra and relative sensitivities of polyphenyls.** P. Bradt and F. L. Mohler (Nat. Bur. of Standards, Washington, D.C., U.S.A.). *J. Res. Nat. Bur. Stand.*, 1958, **60** (2), 143–145.—The mass spectra of pure *m*-quater-, *m*-sexa- and *m*-octophenyl were measured by evaporating the sample from a tube-furnace into the ionisation chamber of a 60° instrument (resolution ≃ 1 in 300) operated at a constant field of 1100 V. The measured ion-currents from a synthetic mixture of *m*-ter-, *m*-quater, *m*-quinque-, *m*-sexa- and *m*-octo-phenyl, evaporated during a stepwise rise of temp. from 125° to 290°, show that the relative sensitivity of ions of polyphenyl mol. increases rapidly with increasing mol. wt. W. J. BAKER

3795. **Determination of purines and pyrimidines by paper chromatography.** V. Blazsek (Med. Univ., Tirgu-Mures, Romania). *Naturwissenschaften*, 1958, **45** (2), 42.—A rapid method consists in spraying the chromatogram with a 0·2% aq. soln. of $CuSO_4$, drying under an infra-red lamp, exposing the paper for 5 min. to ammonia vapour and spraying the paper with a 0·1% ethanolic soln. of diphenylcarbazone. Purines and pyrimidines appear as white areas on a blue background. If the ammonia vapour treatment is omitted, only purines are visible by this method. Amounts of 1 to 10 μg can be determined. E. KAWERAU

3796. **The analytical examination of piperidinomethyl*cyclo*hexanone.** I. Gyenes (Anal. Lab., Pharm. Factory of Kőbánya, Budapest). *Magyar Kém. Foly.*, 1958, **64** (1), 10–16.—Examination of the eutectic mixtures of the hydrochloride of 2-piperidinomethyl*cyclo*hexanone (**I**) with phenacetin or benzanilide showed that the latter mixture was the more sensitive for detecting impurities both by the Kofler and by the capillary m.p. method. The effect of alkaloid reagents on **I**, and the properties of the basic picrate of **I** were studied. The refractive index of **I** was determined. The absorptiometric determination of 50 to 500 μg of **I** by means of *m*-dinitrobenzene is best carried out in 55% pyridine soln.; the optimum conditions are discussed. Any piperidine present can be estimated absorptiometrically, by means of 1:2-naphthaquinone-4-sulphonic acid. The base content of the picrate of **I** can be determined with 0·01 *N* $HClO_4$ in a glacial acetic acid - chlorobenzene - acetic anhydride medium. The hydrochloride of **I** can be titrated with 0·05 *N* $HClO_4$ in glacial acetic acid in the presence of Hg^{II} acetate; if acetylisable amines are present, both Hg^{II} acetate and acetic anhydride must be used. The optimum conditions for the

extraction of **I** from an alkaline medium by means of $CHCl_3$ were studied; after drying the organic solvent, the base can be titrated directly with 0·005 *N* toluene-*p*-sulphonic acid.

A. G. PETO

3797. **Modern trends in petroleum analysis.** H. Powell and W. H. Thomas (British Petroleum Co. Ltd., Sunbury-on-Thames, England). *J. Inst. Petrol.*, 1958, **44**, 19–28.—Recent advances in methods for the examination of petroleum products are reviewed. These show a marked trend towards the use of automatic physical methods of analysis such as the various types of spectrometry, light scattering, polarography, titrimetry, chromatography, thermal diffusion, and viscometry. The computing and statistical analysis of results are also considered briefly. The increased cost of instrumental methods is easily covered by savings in refinery operation. A. JOBLING

3798. **Investigation of aromatic content of kerosine and gas-oil fractions by ultra-violet light absorption.** T. Bálint (Hungarian Res. Inst. for Mineral Oil and Natural Gas, Veszprém). *Acta Chim. Acad. Sci. Hung.*, 1958, **15** (2), 139–150 (in English).—Mono- and bi-cyclic aromatic compounds in petroleum fractions boiling between 180° and 285° can be determined by extinction measurements in the u.v. region. The extinction due to bicyclic compounds is measured at 280 mμ and a correction is made for the deviation caused by 2:6-dimethylnaphthalene. This correction is calculated from an extinction measurement at 324 mμ. The probable relative error is ± 4%. If the content of S is > 0·1%, treatment with $Hg(NO_3)_2$ is necessary. Monocyclic compounds are determined by an extinction measurement at 195 mμ corrected for the extinction due to bicyclic compounds. The mean mol. wt. (obtained from the mean b.p.) must be known. No interfering compounds occur in straight-run distillates. Olefins in cracked fractions interfere slightly. The mean error is about 1%.

G. BURGER

3799. **Use of infra-red absorption spectra in the determination of sulphide sulphur in the asphalt-resin portion of petroleum.** E. A. Glebovskaya and A. P. Shishkova. *Trudy Vses. Neft. Nauch. Geologo-Razved. Inst.*, 1957, (105), 11–22; *Ref. Zhur., Khim.*, 1958, Abstr. No. 915.—Treat the samples with H_2O_2 to oxidise sulphide S and determine the $=SO_2$ groups by the absorption in the region 1120 to 1160 cm^{-1}, with a calibration curve. Prepare standards by dilution of diheptyl sulphone with benzene in the analysis of liquid samples and with petroleum jelly for solids. The layer depth is 0·05 mm for a soln. and 0·01 mm for a jelly. The sensitivity of the detection of S at the long-wave max. of the double band of the $=SO_2$ group is 0·05% for a soln. and 0·25% for a jelly. With concn. of S up to 2% use soln. and for higher concn. use a jelly, in the latter case introducing a correction coeff. for the difference of absorption in the liquid and solid states. Comparative figures are given for the spectrographic and chemical determinations on various samples of petroleum, which agree for the higher fractions but not for the lower fractions. C. D. KOPKIN

3800. **Separate determination of C_1 to C_4 or higher acids in the aqueous condensate obtained in the oxidation of paraffins.** A. M. Zamÿshlyaeva and L. L. Cherno-Ivanova. *Trudy Vses. Nauch. Inst. Zhirov*, 1957, (16), 148–150; *Ref. Zhur., Khim.*, 1958, Abstr. No. 993.—Neutralise the sample of aq. condensate (2 to 3 g) with 0·5 *N* KOH. Filter off any $Fe(OH)_3$ ppt. formed, wash it with warm water and titrate the formic acid in the filtrate and wash liquors with 0·1 *N* $KMnO_4$. Filter off the ppt. obtained during the permanganate titration, wash it with water and evaporate the filtrate and wash liquors to dryness. Take up the residue with 20% H_2SO_4 (10 ml) and extract with benzene (× 3). Titrate the benzene extracts with 0·1 *N* NaOH to determine butyric acid. Saturate the aq. soln. remaining after the extraction of the butyric acid with $(NH_4)_2SO_4$ till a voluminous ppt. is formed and extract with benzene (× 3). Titrate the benzene extracts with 0·1 *N* NaOH to determine propionic acid. Calculate the acetic acid content by the difference between total acid and total formic, propionic and butyric acids. The described method may be used for the analysis of aq. condensates containing ≯ 0·02% of aldehydes, ketones and alcohols; with > 0·02% a correction must be applied. Acids containing more than 4 carbon atoms are extracted with the butyric acid by the benzene, and are calculated as C_4 acids.

C. D. KOPKIN

3801. **Analytical evaluation of higher phenolic fractions of coal tar from the viewpoint of phenol - formaldehyde resin production.** V. Vorobjov (Res. Inst. of Isolants, Bratislava, Czechoslovakia). *Chem. Průmysl*, 1958, **8** (1), 50–51.—The method consists in direct bromination with 0·1 *N* $KBrO_3$ - KBr in acid methanolic medium, the equiv. point being indicated amperometrically with the use of the rotating platinum electrode. The method yields reproducible results, both with pure phenols and technical products; the values obtained from the average functionality (expressed as moles of bromine per mole of phenol homologue) are directly related to the behaviour of raw materials during their reaction with formaldehyde. J. ZÝKA

3802. **Determination of nitrogen in coke by the Dumas - Schöniger method.** R. Belcher and M. K. Bhatty (Birmingham Univ., England). *Fuel, Lond.*, 1958, **37** (2), 159–165.—The Dumas - Schöniger micro-analytical method has been adapted to the semi-micro scale. Finely ground coke (50 mg) is used for each determination, otherwise the procedure is standard. By this method, 16 determinations can be made in an 8-hr. day. Results agree closely (± 0·4%) with the Kjeldahl method with a mercury catalyst, or the Kjeldahl - $KMnO_4$ oxidation, but results obtained by the Kjeldahl method with a selenium catalyst are low.

P. M. SORGO

3803. **Determination of nitrogen in coke by the semi-micro Kjeldahl method.** R. A. Mott and H. C. Wilkinson (British Coke Res. Ass., 74 Grosvenor St., London). *Fuel, Lond.*, 1958, **37** (2), 151–158.—The following method has been devised for routine use in the laboratory. Coke (240 mesh) (100 mg), is heated with 2 g of a mixed catalyst, K_2SO_4 - $HgSO_4$ (7:1, by wt.), and 4 ml of H_2SO_4 (sp. gr. 1·84), until the digest is clear. The subsequent recovery of ammonia is the same as with coal. The times of digesting vary from 1 to 2 hr. for coke with > 1·0% of volatile matter, to 18 hr. for coke with < 0·5% of volatile matter. The presence of selenium in the catalyst reduces nitrogen values owing to decomposition of ammonium compounds during the long digestion required. Although no

comparative values are given for other methods, variations in the amounts of reactants and coke give results with an estimated standard deviation of ± 0·4%. P. M. SORGO

3804. **Determination of total sulphur in solid fuels.** W. Lange and P. Mohrhauer (Hauptlab. Dortmunder Bergbau A.-G., Dortmund, Germany). *BrennstChemie*, 1958, **39**, 55–58.—Comparative tests on 18 samples, including coals, lignites, anthracite and shale, by the German official methods [Eschka (*E*) and combustion (*C*) in contact with $FePO_4$: DIN 51,724], and by reduction with Li metal (*R*) (*cf.* Radmacher and Mohrhauer, *Anal. Abstr.*, 1954, **1**, 2142) show that, on sulphur contents of 0·35 to 13·1%, the percentages found by *E*, *C* and *R* differed by ≯ 0·04, and the respective overall mean values were in the ratio $E:R:C$ = 106:103:100. A. R. PEARSON

3805. **Photometric determination of cyanide in [coal] gas.** A. M. Rozina, N. M. Dankova, N. I. Amitina and E. M. Rutshteĭn. *Koks i Khimiya*, 1957, (5), 45–46; *Ref. Zhur., Khim.*, 1958, Abstr. No. 941.—The method comprises conversion of CN^- into SCN^- with subsequent measurement of the colour produced on addition of $FeCl_3$. Pass 3 to 3·5 litres of gas in 0·5 hr. through two Petri flasks containing 25 ml each of ≃ 4 *N* NaOH. Transfer the NaOH soln. to a 100-ml flask and make up to the mark with water. To 50 ml add ammonium polysulphide (0·2 ml) and 10% $CdCl_2$ soln. (5 ml) and boil for 2 min. Cool, filter into a 100-ml flask, wash the residue with small amounts of water and acidify with 10 ml of ≃ 60% HNO_3. Cool, add 10% $FeCl_3$ soln. (5 ml) and make up to the mark with water. After 0·5 hr. measure the absorption with a green filter against a blank. Compare with a calibration curve. The described method is not less accurate than other known methods and is more rapid, requiring ≃ 1·5 hr. C. D. KOPKIN

3806. **Colorimetric determination of iron in gelatin with 2:2′-dipyridyl.** J. Beneš (Inst. Phys. Chem., High School of Chem. Technol., Prague). *Chem. Průmysl*, 1958, **8** (2), 84–85.—*Procedure*—Carefully ignite the sample of dried gelatin (2 g) in a porcelain crucible, add conc. HNO_3 (1 ml for 1 g of the sample), evaporate and ignite in a furnace at 450°. Dissolve the residue in conc. HNO_3 (2 ml) and evaporate to dryness. Dissolve the residue in 0·25 *N* HCl and dilute to 50 ml. To 25 ml add hydroxylamine sulphate soln. (10%) (5 ml) and adjust to pH 4 by adding 2 *M* Na acetate. Add 2:2′-dipyridyl (0·1%) (3 ml), mix and set aside for 30 min. Measure the extinction with the use of a blue - green filter. J. ZÝKA

3807. **Colorimetric determination of thymol in thyme oil.** L. Fibranz, M. I. Blake and C. E. Miller (Sch. of Pharm., North Dakota Agric. Coll., Fargo). *J. Amer. Pharm. Ass., Sci. Ed.*, 1958, **47** (2), 133–135.—Mix a soln. of the sample in $CHCl_3$ (1 ml containing ≃ 80 μg of oil) with a 0·125% soln. of *p*-dimethylaminobenzaldehyde in conc. H_2SO_4 (5 ml), shake for 10 sec. and measure the extinction with the use of a blue filter (400 to 465 mμ). Calculate the concn. of thymol by reference to a calibration curve. The precision is ≃ ± 1%. The results for samples of the red and white varieties of thyme oil obtained by this method agree with those by the U.S. National Formulary X assay procedure and a non-aq. titration method. Linalol, α-pinene, *p*-cymene, geraniol and borneol produce colour with the reagent, but are normally present in the oil in such small concn. that interference with the determination of thymol is negligible. A. R. ROGERS

3808. **Paper-chromatographic analyses of some intermediate products for the dyestuff industry.** S. Hoffman, V. Paul and B. Jacob. *Rev. Chim., Bucharest*, 1958, **9** (1), 44–46.—The separation of 1-naphthylamine-3:6:8-trisulphonic acid (**I**) from 8-amino-1-naphthol-3:6-disulphonic acid is achieved by using aq. soln. of their sodium salts (1% at pH 6). The 15-cm filter-papers used are perforated in the centre, at which point the test sample is applied, and are enclosed in Petri dishes during the experiment, which is carried out at 20° to 25°. The sample is applied in stages, drying between each stage so that the diameter of the spot does not exceed 1·5 cm. A soln. of NaCl (10%), applied at the centre of the paper, is used as developer. After development for 1·5 hr., a 10 to 12-cm chromatogram is obtained, which is examined under filtered ultra-violet light. The effect of **I** is made clearer by a short exposure of the chromatogram to ammonia vapour, which changes the blue - violet fluorescence to an intense yellow. The same method is used for separation of 1-naphthol-3:6:8-trisulphonic acid from chromotropic acid. The following method for the separation of 1-naphthylamine-4-sulphonic acid from 1-naphthylamine-5-sulphonic acid, by using aq. soln. of their sodium salts (1% at pH 7), is employed. Whatman No. 1 paper (25 cm × 15 cm) is used in a closed chamber, and the 0·0015-ml portions of the soln. containing 15 μg of the substance are placed 2 cm from the base of the paper, 3 cm apart. The spots (0·7 cm) obtained are dried, and then kept for 20 hr. in a chamber containing 4 crucibles of solvent (*n*-butanol - acetic acid - water, 4:1:5). The paper is then dipped (0·5 cm) vertically into NaCl soln. (10%) and developed for about 2·5 hr. at 23° to 24°, the solvent having risen 23 cm. Results are examined under filtered ultra-violet light. H. SHER

3809. **Titration of triphenylmethane dyes with vanadium(II) sulphate in the presence of sodium xylenesulphonate.** M. Matrka and Z. Ságner (Res. Inst. Org. Synth., Pardubice-Rybitví, Czechoslovakia). *Chem. Průmysl*, 1958, **8** (1), 22–24.—In this reductimetric determination of triphenylmethane dyes, a soln. of Na *m*-xylenesulphonate was substituted for ethanol used previously as a solvent for the leuco base formed during the titration. *Procedure*—Weigh 0·3 to 0·4 g of basic dyestuff or 0·6 to 1 g of acid dyestuff and dissolve in water (100 ml) (for the acid sample) or in a 15% soln. of Na *m*-xylenesulphonate (100 ml) (for the basic sample), add 50 ml of satd. buffer soln. (K Na tartrate, Na citrate and Na H tartrate). Warm to 50° and titrate with 0·1 *N* VSO_4 in an atmosphere of N. The end-point can be determined visually or with potentiometric control. Twelve dyestuffs were determined with good accuracy and the proposed method was compared with the indirect titanimetric titration. J. ZÝKA

3810. **Determination of sulphate acid ester in unstabilised cellulose nitrate by the hydrolysis method.** P. E. Gagnon, K. F. Keirstead and B. T. Newbold (Dept. Chem., Laval Univ., Quebec, Canada). *Canad. J. Chem.*, 1958, **36** (2), 291–295.—In a previously described method for determining

the sulphate ester, the method of titration in acetone - water solution before and after hydrolysis was used (*cf.* Gagnon *et al.*, *Anal. Abstr.*, 1957, **5**, 167). The zero value was obtained by extrapolation of the titration value to zero time. This gives results that are too high. In the present evaluation of this method, a moist sample of cotton cellulose nitrate (**I**), or wood cellulose nitrate (**II**), is dissolved in 90% acetone - water, which prevents hydrolysis, and the acidity is determined with 0·01 *N* KOH, with a pH meter. The error obtained by extrapolation was as high as 65% for **I**, and 50% for **II**. The volumetric method is based on a 24-hr. hydrolysis of the cellulose nitrate sulphate ester. Comparison of the results with those from a 48-hr. titre shows that hydrolysis continues slowly, giving up to a 5% increase in the sulphate value. A comparison between gravimetric and volumetric sulphate percentages, after 24 hr., gives a standard deviation of 8·2 for **I**, and 4·3 for **II**, which is less than the difference between 24- and 48-hr. titres. In duplicate determinations with **I** and **II** containing about 100% of sulphate ester, the standard deviations are 3·0 and 4·4, respectively. With a sulphate ester content of 69 to 43% for **I**, and 69 to 47% for **II**, the standard deviations rise to 5·8 and 10·3, respectively. P. M. SORGO

3811. **Turbidimetric titration of polymer solutions.** F. Krašovec, N. Vene and A. Peterlin ("J. Stefan" Inst., Ljubljana, Yugoslavia). *Rep. "J. Stefan" Inst., Ljubljana*, 1957, **4**, 165–173 (in English).—The mol.-wt. distribution of polymers in dil. soln. can be determined by measurement of the turbidity during the slow addition of a precipitant. For poly(vinyl chloride) a suitable solvent is *cyclo*hexanone and the precipitant a mixture of light petroleum and CCl_4. Once solubility curves have been obtained for homogeneous fractions, the vol. of added precipitant enables the mol. wt. of the fraction pptd. to be determined and the turbidity is a measure of the amount pptd. A correction for the error caused by change of particle size is obtained by measuring the ratio of the intensities of scattered light at angles of 45° and 135°. A correction is made for light reflected at interfaces. G. BURGER

3812. **Infra-red spectra of thermally degraded poly(vinyl chloride).** R. R. Stromberg, S. Straus and B. G. Achhammer (Nat. Bureau of Standards, Washington, D.C.). *J. Res. Nat. Bur. Stand.*, 1958, **60** (2), 147–151.—Infra-red spectra of three specimens of poly(vinyl chloride) before and during pyrolysis are recorded. N. E.

3813. **Polarographic determination of benzoyl peroxide in certain plastics.** V. D. Bezuglŷĭ and V. N. Dmitrieva (Kharkov Works of Dental Materials). *Zhur. Prikl. Khim.*, 1958, **31** (2), 298–305.—In a mixture of methanol and benzene (4:1), containing 2% of NH_4NO_3 and also methyl red as maximum suppressor, benzoyl peroxide is irreversibly reduced on the dropping mercury electrode showing $E_{\frac{1}{2}}$ of + 0·27 V *vs.* the S.C.E. For determination of benzoyl peroxide in emulsion-polymerised methyl methacrylate, the sample (1·5 to 1·8 g) is dissolved in 20 ml of benzene, the polymer is pptd. by addition of methanol, 2 g of NH_4NO_3 and 1 ml of 0·2% methyl red soln. are added, and the soln. is diluted to 100 ml. After 20 min., 10 ml is removed and freed from O, and the polarogram over the range + 0·45 to − 0·2 V is obtained.
G. S. SMITH

3814. **Determination of zinc in rubber.** F. Oehlmann (Dtsch. Amt f. Material u. Warenprüfung, Halle, Saale). *Chem. Prùmysl*, 1958, **8** (1), 44–46.—A modification of the Urbasch method (*Chemiker-Ztg*, 1922, **46**, 54) is described. The rubber is burned, the ash is extracted with hot HCl, and Zn is quant. separated from the soln. on an ion-exchange column of Wofatit L150. The adsorbed Zn is eluted with 0·05 *M* HCl and determined in the eluate titrimetrically with $K_4Fe(CN)_6$ soln., according to Urbasch. J. ZÝKA

3815. **Colorimetric determination of ferric oxide in rubber and vulcanisates.** L. L. Bogina and I. P. Martyukhina. *Leghaya Prom.*, 1957, (6), 31–32; *Ref. Zhur., Khim.*, 1958, Abstr. No. 7607.—The described method is based on the use of sulphosalicylic acid (**I**). Ash 1 g of material, add to the residue 20 ml of hot HCl (1:3) and 10 to 15 ml of hot water, boil till the iron oxides are completely dissolved, filter the soln. and wash the filter 3 or 4 times with hot water. To the filtrate and wash liquors add 10 ml of a 5% NH_4Cl soln. and 25 ml of a 10% **I** soln. and neutralise first with a few drops of 40% NaOH soln., followed by 0·5 *N* NaOH till the soln. is yellow. Dilute the soln. to 100 ml and after 15 min. measure the light absorption with a green filter, with water as standard. Find the content of Fe from a calibration curve. The presence of small quantities of Mn, Al and Ca does not cause interference; Cu interferes.
C. D. KOPKIN

3816. **Quantitative determination of antioxidants [in rubber], after chromatographic separation on completely acetylated filter-paper. III. Determination of *p*-(*p*′-tolylsulphonylamino)phenyl-*p*-toluidine, NN′-di-*o*-tolylethylenediamine and quinol monobenzyl ether [*p*-benzyloxyphenol].** J. W. H. Zijp (Rubber Res. Inst., T.N.O., Delft). *Rec. Trav. Chim. Pays-Bas*, 1958, **77** (2), 129–132 (in English).—*p*-(*p*′-Tolylsulphonylamino)phenyl-*p*-toluidine (**I**), NN′-di-*o*-tolylethylenediamine (**II**) and *p*-benzyloxyphenol (**III**) are extracted from vulcanised rubber with benzene, and separated by paper chromatography on completely acetylated filter-paper, **I** with benzene - ethanol (1:1) as mobile phase followed by elution with benzene, **II** with benzene - ethanol (1:1) followed by ethanol elution, and **III** with ethanol - benzene - water (4:1:1) followed by ethanol elution. For the colorimetric determination, the separated **I** is oxidised with 2:4-dichlorobenzoyl peroxide, and **II** and **III** are coupled with diazotised sulphanilic acid, and the extinctions of the products are measured. M. DAVIS

3817. **Infra-red quantitative analysis data.** *Anal. Chem.*, 1958, **30** (2), 304–305.—The following data have been published under the co-operative programme between *Analytical Chemistry* and the Coblentz Society of the U.S.A. **Determination of pentan-2-ol in mixture with 2-bromopentane plus 3-bromopentane,** W. H. Washburn and M. J. Mahoney. **Determination of toluene in 1-chloropent-1-en-3-one,** W. H. Washburn and F. A. Scheske. **Determination of 1-chloropent-1-en-3-one in 1-chloro-3-ethylpent-1-en-4-yn-3-ol,** W. H. Washburn and F. A. Scheske. **Determination of chlorcyclizine and pramoxine in mixtures,** W. H. Washburn. **Determination of 2-, 3- and 4-ethyltoluene in ethyltoluene mixtures,** L. R. Kiley. **Analysis of ethane, propane, *iso*butane and *n*-butane,** R. C. Wilkerson, M. C. Harvey and R. M. Guedin. **Analysis of phenol, *cyclo*hexene, *cyclo*hexanone, *cyclo*hexanol**

mixtures, R. T. Scheddel. **Determination of N-(β-hydroxyethyl)phenylacetamide in presence of ethanolamine and ethyl phenylacetate,** W. H. Washburn.

See also Abstracts—**3587**, Identification of organic bases. **3619**, Determination of Cu and As in carbon blacks. **3683**, Micro-determination of As in coal and coke. **3759**, Determination of mineral matter in coal. **3825**, Determination of pentachlorophenol. **3858**, Determination of *p*-hydroxyanilinoacetic acid. **3957**, Differential thermal analysis of organic solids.

4.—BIOCHEMISTRY
INCLUDING DRUGS, FOOD, SANITATION, AGRICULTURE
Blood, Bile, Urine, etc.

3818. **Rapid system of microchemical analysis for the clinical laboratory.** A. Saifer, S. Gerstenfeld and M. C. Zymaris (Isaac Albert Res. Inst., Jewish Chron. Dis. Hosp., Brooklyn, N.Y., U.S.A.). *Clin. Chem.*, 1958, **4** (2), 127–141.—In the proposed system, heparinised plasma, collected in silicone-treated tubes, is used in place of serum or whole blood; vol. of sample, standards and blanks are measured by calibrated pipette tip burette technique; decantation is used for quant. transfer from centrifuged ppt.; automatic syringe pipettes are used in place of volumetric pipettes, and enzyme reactions are employed whenever feasible.
H. F. W. KIRKPATRICK

3819. **Determination of calcium in serum.** W. Pilz (Med. Div., Bayer A.-G., Leverkusen, Germany). *Klin. Wochschr.*, 1958, **36** (4), 181–183.—Mix the serum (2 ml) in a centrifuge tube with 2% ammonium oxalate soln. (1 ml). After 10 min. centrifuge the soln. for 10 min. at 3000 r.p.m. and drain the ppt. on filter-paper for 5 min. To the ppt. add H_2O (3 ml) and HCl (2 drops), mix, and pour into a 50-ml wide-mouth Erlenmeyer flask, rinsing with H_2O to give a total vol. of $\simeq$ 20 ml. To the soln. add 0·001 *M* EDTA (6 to 7 ml), aq. NH_3 (8 to 12 drops), and a little indicator powder (1% of Eriochrome black T in NaCl). Titrate the excess of EDTA with 0·001 *M* $ZnSO_4$ from green - blue to violet. The error due to Mg in this method is $< +$ 0·0001% of Ca.
H. F. W. KIRKPATRICK

3820. **Complexometric titration of micro and ultra-micro quantities of calcium in blood serum, urine, and inorganic salt solutions.** B. N. Bachra, A. Dauer and A. E. Sobel (Jewish Hosp. of Brooklyn, N.Y., U.S.A.). *Clin. Chem.*, 1958, **4** (2), 107–119.—A micro-adaptation of the method of Patton and Reeder (*Anal. Chem.*, 1956, **28**, 1026) is described.
H. F. W. KIRKPATRICK

3821. **Electrometric method for the determination of chloride in serum and other biological fluids.** D. Seligson, G. J. McCormick and K. Sleeman (Grad. Sch. of Med., Univ. of Pennsylvania, Philadelphia, U.S.A.). *Clin. Chem.*, 1958, **4** (2), 159–169.—The method of Cunningham *et al.* (*J. Biol. Chem.*, 1941, **139**, 11) has been improved by the use of a special titrating table which shortens the equilibrium time for additions of $AgNO_3$ from 30 to 2 to 3 sec. Accuracy and speed are enhanced by the use of a Scholander micro-burette and a special electrical circuit.
H. F. W. KIRKPATRICK

3822. **Determination of mercury in urine.** S. Nobel and D. Nobel (Monmouth Mem. Hosp., Long Beach, N.J., U.S.A.). *Clin. Chem.*, 1958, **4** (2), 150–158.—Mercury in urine is reduced to the metallic form by a Cu - hydrazine system, oxidised to Hg^{2+} by H_2SO_4 and $KMnO_4$ at room temp., excess of $KMnO_4$ is removed with hydroxylamine hydrochloride and the Hg^{2+} are titrated with dithizone. *Procedure*—Measure the urine (50 ml) into a 250-ml glass-stoppered Erlenmeyer flask and add, swirling after each addition, 15 *N* H_2SO_4 (0·5 ml), 0·39% $CuSO_4.5H_2O$ soln. (1 ml), and (without touching the neck of the flask) 95% hydrazine hydrate (0·2 ml). Stopper the flask, set it aside for 10 min., add 10 *N* NaOH (2·0 ml) and swirl the flask for 20 sec. Add 15 *N* H_2SO_4 (9 ml), octan-2-ol (2 drops), swirl again; add 6% $KMnO_4$ soln. (35 ml) slowly, washing the side of the flask, stopper loosely and set it aside at room temp. for 1 hr. Decolorise with 20% hydroxylamine hydrochloride (1-ml additions), stopper the flask loosely and set it aside for 30 min. Pour the soln. into a 300-ml separating funnel, rinsing the flask with H_2O (2 × 15 ml). Shake with $CHCl_3$ (5 ml) and discard the $CHCl_3$, titrate by extraction with dithizone soln. (5·5 mg per litre in $CHCl_3$) to the nearest 0·5 ml. Prepare a standard curve by diluting aliquots (0 to 2·0 ml) of standard soln. (1 ml $\equiv$ 10 μg of Hg) to 50 ml with H_2O and carrying through the process described above.
H. F. W. KIRKPATRICK

3823. **Determination of iron in biological material by spectrophotometry of ferric perchlorate.** I. D. P. Wootton (Postgrad. Med. Sch., London). *Biochem. J.*, 1958, **68** (2), 197–199.—Iron in the range of 0·02 to 1 mg per 100 ml is determined by digestion of the sample with HNO_3 - $HClO_4$ and spectrophotometric measurement of the resulting $Fe(ClO_4)_3$ at 240 mμ. Interference from other ions is usually negligible. Perchlorates of V and Mo have u.v. absorptions comparable with that of $Fe(ClO_4)_3$; of other elements, only Cu is likely to interfere, at least in animal specimens. Interference can then be reduced by measurement at 260 mμ, with some sacrifice of sensitivity. The method is claimed to be rapid and precise.
J. N. ASHLEY

3824. **Spectrophotometric determination of iron in serum.** Shunji Umemoto and Yasuhisa Yamamoto (Balneological Lab., Okayama Univ., Misasa Hot Springs, Tottori Prefecture, Japan). *Bull. Chem. Soc. Japan*, 1958, **31** (1), 1–3.—Torii's method of determining ferrous iron with *m*-methoxy-*o*-nitrosophenol (**I**) (*cf. Anal. Abstr.*, 1956, **3**, 115) is applied to the determination of Fe in human blood serum. Clear serum (2·0 ml) is acidified with 6 *N* HCl (0·5 ml), set aside for 10 min., treated with 20% trichloroacetic acid (1·0 ml) and centrifuged. The liquid is filtered if necessary, and the residue is washed with 1 ml of water and 0·5 ml of 20% trichloroacetic acid. The combined liquids are reduced in a stoppered flask with 10% hydroxylamine hydrochloride soln. (0·5 ml) for 15 min., with occasional shaking, and neutralised with 6 *N* aq. NH_3 to *p*-nitrophenol soln. (1 drop of 0·1%). Acetic acid - Na acetate buffer (pH 4·5) (2 ml) is added, followed by 5% $Na_2S_2O_3$ soln. (0·5 ml) to remove Cu, and, after 5 min., satd. aq. **I** (1·0 ml). The vol. is adjusted with water to 10 ml, and, after 15 min., excess of **I** is removed by extraction with CCl_4 (4 × 2 ml). The aq. soln. is centrifuged to remove drops of CCl_4, and the extinction is measured at 700 mμ. The calibration is linear between 0 and 800 μg of Fe per ml. Recoveries with known additions of Fe are within 2%.
A. B. DENSHAM

3825. **Toxicological detection and determination of pentachlorophenol.** K. Erne (State Vet. Med. Inst., Stockholm, Sweden). *Acta Pharm. Tox., Kbh.*, 1958, **14** (2), 158–172.—The sample of biological material (10 g) is dried with anhyd. Na_2SO_4 and extracted with diethyl ether in a Soxhlet apparatus. The dry ether extract, dissolved in CCl_4, is passed through a column of Celite treated with fuming H_2SO_4; the eluate containing the pentachlorophenol (**I**) is concentrated and extracted with 5% aq. $Na_4P_2O_7$. If spectrophotometric examination of the aq. extract shows the presence of **I**, a portion of it is acidified, extracted with diethyl ether and the ether extract purified by partition chromatography on paper. After development, the area containing **I** is extracted quant. with methanol and the **I** is determined from the extinction at 324 mμ, corrected for irrelevant absorption by a base-line technique. As little as 1 to 2 μg of **I** per g of sample is detectable and concn. of ≮ 5 μg can be determined. The recovery is > 85% and the reproducibility is ± 7% of the mean.
W. H. C. Shaw

3826. **Detection and determination of chlorpromazine (Largactil) in biological liquids.** C. Citterio and F. Mattei (Osped. Psichiat. Prov., Rome). *Law. Psichiat.*, 1957, **20**, 189–193.—The method of Dubost and Pascal (*cf. Anal. Abstr.*, 1955, **2**, 2213) is modified as follows. For blood or cerebrospinal fluid add 4 ml of conc. HCl and 1 ml of H_2O to 5 ml of oxalated blood, warm in steam for 5 min., cool, dilute to 20 ml, neutralise with 50% NaOH soln., add 20 ml of diethyl ether, shake for 5 min., separate the ether layer, repeat the operation three times and collect the ether fractions. Wash the extracts twice with 10 ml of 0·2 *N* NaOH and once with 10 ml of H_2O, shake with 16 ml of 0·1 *N* H_2SO_4, separate the H_2SO_4 phase, subject to vacuum, and warm to 55° to 60° to expel ether. To 2 ml add 0·2 ml of conc. HNO_3 (*d* 1·4) and read the light absorption at 514 mμ in comparison with a blank made with H_2O, and calculate in comparison with standards obtained with 0·1 to 1·0 ml of 0·02% chlorpromazine soln. For urine, take 20 ml of urine, add 16 ml of conc. HCl (*d* 1·19) and 4 ml of H_2O, boil, then cool, neutralise with 50% NaOH soln., add 3 ml of 20% Na_2CO_3 soln., and continue with diethyl ether extraction, etc., as for blood. Chem. Abstr.

3827. **Method for determining 1-butyl-3-*p*-tolylsulphonylurea (tolbutamide) in human blood serum.** E. Bladh and Å. Nordén (Univ. Hosp., Lund, Sweden). *Acta Pharm. Tox., Kbh.*, 1958, **14** (2), 188–194.—In the method described, the serum (1 ml) is diluted, acidified and extracted with $CHCl_3$. After separation and purification by passing the $CHCl_3$ soln. through a column of $CaCO_3$ or of $NaHCO_3$, the residue from the $CHCl_3$ eluate is dissolved in ethanol and tolbutamide is determined from the extinction at 228 mμ. Blank values equivalent to 3·9 ± 0·6 mg % of tolbutamide were found. Recovery from serum was 103 ± 5%.
W. H. C. Shaw

3828. **Determination of 2-methylpentane-2:4-diol ("hexylene glycol") in the urine of man and rats.** E. Jacobsen (Medicinalco Ltd., Copenhagen, Denmark). *Acta Pharm. Tox., Kbh.*, 1958, **14** (2), 195–208.—The method is based on the development of turbidity when aq. soln. of 2-methylpentane-2:4-diol (**I**) are heated with dil. HCl. The apparent extinction at 1075 mμ under standard conditions is related to concn. between 0·05 and 1·50 mg of sample per ml. Interference by urine pigments is eliminated by addition of **I** at two levels to the sample, and calculation of the results by the method described. Direct application of the method gives total **I**; free **I** may be determined after distillation *in vacuo* (recovery 70 to 100%). The standard deviation for a single determination is 0·05 mg per ml for human and 0·8 mg per ml for rat urine.
W. H. C. Shaw

3829. **Comparison of direct and extraction methods for the determination of T-1824 (Azovan blue) in plasma and serum.** J. F. Murray and J. P. Shillingford (Postgrad. Med. Sch., London). *J. Clin. Path.*, 1958, **11** (2), 170–172.—Direct determination is liable to considerable error when haemolysis or lipaemia is present. Acetone extraction is more satisfactory in giving consistent results.
H. F. W. Kirkpatrick

3830. **Simplified colorimetric method for true blood glucose.** G. Athanail and P. G. Cabaud (Brooklyn Hosp., N.Y., U.S.A.). *J. Lab. Clin. Med.*, 1958, **51** (2), 321–324.—The method is based on reaction of glucose with 2-aminodiphenyl; other reducing substances in blood do not interfere. *Reagent*—Prepare a 0·4% soln. of 2-aminodiphenyl in glacial acetic acid. Allow to stand in the dark for 2 to 3 days before use and store in the dark. *Procedure*—Prepare either a tungstic acid (Folin and Wu) or a $Ba(OH)_2$ - $ZnSO_4$ (Somogyi) filtrate of the blood sample so that the final dilution is 1 in 10. Place duplicate 1-ml amounts of the filtrate, containing 0·01 to 0·5 mg of glucose, in test-tubes (150 mm × 16 mm) and add 5 ml of reagent to each tube. Place in a boiling-water bath for 45 min., cool, and measure the extinction at 650 mμ against a blank prepared with 1 ml of water in place of the sample filtrate. Calibrate with 1-ml portions of anhyd. glucose soln. (1 to 50 mg per 100 ml).
W. H. C. Shaw

3831. **Determination of uric acid in biological materials.** A. E. Steel (Royal Bath Hosp., Harrogate, England). *Biochem. J.*, 1958, **68** (2), 306–309.—The method, which is based on the reaction between uric acid and tungstophosphoric acid in the presence of NaCN, can be used for the determination of uric acid in as little as 0·05 ml of serum containing up to 25 mg of the acid per 100 ml. The reaction is carried out at 20° and the extraction of the blue soln. is measured spectrophotometrically at 690 mμ. Several substances modify the colour reaction; the most important of these, in the assay of serum, are amino acids. Dilution of the test soln. reduces this effect and yields higher results.
J. N. Ashley

3832. **Determination of phylloerythrin in blood.** D. D. Perrin (Ruakura Animal Res. Sta., Hamilton, N.Z.). *Biochem. J.*, 1958, **68** (2), 314–318.—Phylloerythrin in blood is extracted into acetic acid - diethyl ether, then distributed between diethyl ether and dil. HCl, and is determined spectrophotometrically in diethyl ether at one or other of the peaks in the banded absorption spectrum in the visible and near u.v. regions. The method, which appears to be applicable to other biological material, especially urine, enables 0·05 to 3·5 mg per 100 ml of plasma to be determined; a modification is also described which gives approx. values down to 0·005 mg per 100 ml. Protoporphyrin and other porphyrins do not interfere significantly.
J. N. Ashley

3833. **Paper electrophoresis of urinary porphyrins.** N. Verghese (Dept. of Biochem., Madras Med. Coll., India). *J. Clin. Path.*, 1958, **11** (2), 191–192.—Separation and determination of the different porphyrins excreted in porphyria may be made as follows. Collect a 24-hr. urine with toluene as preservative, acidify 500 ml with glacial acetic acid to pH 4, allow to stand for 24 hr. under a layer of toluene, and filter. Wash the brown ppt. of porphyrins with H_2O until the washings are neutral to litmus, dissolve in barbitone buffer (5 to 8 ml) as used for serum electrophoresis, by pouring repeatedly through the paper, and carry out the electrophoresis as for serum, with Whatman 3MM filter-paper. Dry the paper at 60° to 70° and examine in u.v. light; porphyrins show as bands of red fluorescence, the area and intensity corresponding approximately to the amount present. For identification, purified porphyrins are spotted as markers and run parallel to the test soln.

H. F. W. KIRKPATRICK

3834. **Factors affecting the rate of coupling of bilirubin and conjugated bilirubin in the Van den Bergh reaction.** G. H. Lathe and C. R. J. Ruthven (Queen Charlotte's Maternity Hosp., London). *J. Clin. Path.*, 1958, **11** (2), 155–161.—The effects of pH, solvent and protein are described and a method is recommended for determining bilirubin in infant's blood. *Procedure*—Add heparinised plasma (0·2 ml) to H_2O (5·4 ml) and transfer 2·8 ml to a second tube for the blank. To the test add freshly prepared diazo reagent (10 ml of 1% sulphanilic acid in 0·2 *N* HCl mixed with 0·3 ml of 0·5% $NaNO_2$ soln.) (0·7 ml) and to the blank add sulphanilic acid soln. alone (0·7 ml). Mix and read the absorption after 5 min., with a green filter. Calculate the conjugated (direct) bilirubin from $[(E_{test} - E_{blank})/E_{standard}] \times 10{\cdot}5$. To each tube add 3·5 ml of methanol, mix and read after 5 min. Calculate the total pigment as before, with the factor × 20. Prepare a standard by refluxing bilirubin (10 mg) with $CHCl_3$ until dissolved and adjusting the vol. to 100 ml. Keep in the dark in a refrigerator. Add the standard soln. (0·2 ml) to methanol (3·5 ml), mix with diazo reagent (0·7 ml) and H_2O (2·6 ml). Read after 5 min. against H_2O.

H. F. W. KIRKPATRICK

3835. **Microbiological assay of vitamins in clinical chemistry.** H. Sobotka (Mt. Sinai Hosp., New York, N.Y., U.S.A.). *Clin. Chem.*, 1958, **4** (2), 93–106.—An account of investigational work in this field is presented. As examples of methods and clinical significance of results, special attention is given to assays of vitamin B_{12} and folic acid.

H. F. W. KIRKPATRICK

3836. **Determination of vitamin A in small amounts of plasma.** Tetsuzo Takahashi and Kazue Kono. *Vitamins, Japan*, 1956, **10**, 350–353.—By the use of the small vessel of the photo-electric colorimeter for small amounts of plasma (0·5 to 1·0 ml), vitamin-A determinations by Takahashi's method (*cf. Vitamins, Japan*, 1952, **5**, 350) gave satisfactory results. The concn. of vitamin A and carotene in 100 ml of adults' plasma were 139 ± 32 i.u. for the former, and 69 ± 38 μg for the latter, those for infants were 113 i.u. and 33 μg, respectively, and those for children were 126 i.u. and 74 μg, respectively. CHEM. ABSTR.

3837. **Photolytic micro-determination of vitamin A in blood serum.** Koji Kimura (Kitasato Inst.). *Vitamins, Japan*, 1956, **11**, 362–368.—Some improvements in the photolytic micro-determination of vitamin A in serum (*cf.* Bessey *et al.*, *J. Biol. Chem.*, 1946, **166**, 177) have been made. These comprise the purification of kerosine and the demonstration of the need for correcting the increased blank optical density due to u.v. irradiation. Vitamin-A ester showed behaviour similar to that of vitamin-A alcohol in the procedure. The values for human-blood-serum vitamin A determined photolytically by this method and those by a glycerol-dichlorohydrin micro-method were almost the same.

CHEM. ABSTR.

3838. **Quantitative determination of vitamin B_{12} in blood serum with *Escherichia coli*.** L. A. Maĭ and L. I. Yavorkovskiĭ, with E. Ya. Krumin' (Republican Blood Transfusion Station, Latvian SSR). *Biokhimiya*, 1958, **23** (2), 237–243.—With *Esch. coli*, mutant 113-3, 20 to 10,000 μμg per litre of vitamin B_{12} in blood serum can be determined, the optimum concn. being 10 or 20 to 75 or 100 μμg per litre. Generally a tenfold dilution gives this concn., 0·1 to 5·0 ml of diluted and treated serum being taken, usually 0·5 to 2·0 ml being convenient. Proteins are pptd. by boiling the sample in closed tubes with acetate buffer soln. (acetate buffer soln. of pH 4·63 - water, 1 : 1) (0·025 to 0·1 ml per 10 ml). To stabilise the vitamin during this treatment, the serum is diluted not with water, but with KCN soln. (1·2 μg per ml). The growth medium is the glucose - salt soln. of Bukin *et al.* (*Biokhimiya*, 1954, **19**, 713); darkening of the medium on repeated stabilisation is avoided by separate preparation and sterilisation of soln. of the two components. The use of a double-strength medium is not recommended. As control to allow for the effect of methionine, a sample of serum is boiled at pH 12 to 13 for 15 to 20 min. to destroy vitamin B_{12}; the residual activity is 3 to 38% of the total, average 14·8% (52 determinations). No relationship was observed between total and residual activity. A standard curve is constructed by measuring the cloudiness obtained from samples with known contents of pure cryst. vitamin B_{12}. It is almost logarithmic in the region 10 or 15 to 75 or 100 μμg, this being the most reproducible and accurate region. Different curves may be obtained at different times and in different laboratories. Occasionally the growth of the micro-organisms is much decreased or even absent, the cause not being known. Discrepancies between parallel determinations do not as a rule exceed 10%. The accuracy of the determination is ± 15%, the sensitivity 0·5 μμg per ml. C. D. KOPKIN

3839. **Prothrombin measurement by two-stage technique with haemolysed whole-blood thromboplastin.** O. H. Dreskin (Jewish Hosp., Cincinnati, Ohio, U.S.A.). *J. Lab. Clin. Med.*, 1958, **51** (2), 312–316.—A mixture of the sample plasma, washed blood cells and Ca^{2+} is incubated at 37°. At suitable time intervals aliquots of the mixture are added to a fibrinogen solution prepared from oxalated plasma by $BaSO_4$ treatment. The observed clotting time is converted to prothrombin units by the table given. Results are compared with those obtained by conventional one- and two-stage prothrombin methods. W. H. C. SHAW

3840. **Quantitative determination of raffinose or melibiose by paper chromatography.** A. Bevenue and K. T. Williams (West Reg. Res. Lab., Albany, Calif., U.S.A.). *Arch. Biochem. Biophys.*, 1958, **73** (2), 291–295.—Separation of the two carbohydrates is by the descending technique on Whatman No. 1

paper with *n*-propanol - ethyl acetate - water (7:1:2). After development for 18 hr. at 25° the chromatograms are air-dried for 30 to 60 min. The strip containing the separated carbohydrates is cut out, dipped in reagent (*i*) or (*ii*) described below, air-dried for 20 to 30 min. and then heated in a forced-air oven at 85° for 10 min. Density measurements are made with 515-mμ light, and a standard curve is prepared on semi-logarithmic paper with 20 to 75 μg of carbohydrate in 5 ml of test solution. Replication on one chromatogram is $\pm$ 5%. *Reagents*—(*i*) Dissolve 0·5 g of Genochrome (*p*-amino-NN-diethylaniline - sulphur dioxide complex) in 50 ml of 95% ethanol and mix with 50 ml of 95% ethanol containing 2 ml of 85% H_3PO_4. The reagent is stable for 1 week when refrigerated. (*ii*) Saturate a 1% soln. of *p*-amino-NN-diethylaniline in 95% ethanol with SO_2 for 2 hr. Add 1 ml of 85% H_3PO_4 to 50 ml of the solution. Prepare fresh daily. W. H. C. SHAW

3841. **Quantitative estimation of sialic acids. III. An anion-exchange-resin method.** L. Svennerholm (Dept. of Med. Biochem., Univ. of Gothenburg, Sweden). *Acta Chem. Scand.*, 1958, **12** (3), 547–554.—Samples of 25 to 200 μg of sialic acids or of tissues and body fluids containing sialic acids are hydrolysed for various times and temp. with various concn. of H_2SO_4 shown to be optimum for different cases (0·5 to 1 *N* H_2SO_4 at 70° to 90° for 1 to 3 hr.), and passed through a Dowex-2 (acetate) column. When more than 0·1 milli-equiv. of inorganic cations is present, the soln. is passed through a Dowex-50 (H) column first. After the column has been washed, the sialic acid is eluted with *M* acetic acid - Na acetate buffer (pH 4·6), and determined colorimetrically by heating with resorcinol reagent (0·2 g of resorcinol, 80 ml of conc. HCl, 0·25 ml of 0·1 *M* $CuSO_4$ and H_2O to 100 ml), and extracting the colour produced with *iso*-amyl alcohol. The absorption is measured at 580 mμ and also at 450 mμ to correct for ribose deriv. present. Other interfering impurities have been removed by the ion-exchange procedure. The recovery is about 95%, but lower in some cases. The results for several analyses of tissue and body fluids are given. The figures given are calculated as N-acetylsialic acid, and it is pointed out that N-glycollylsialic acid, which is likely to be present in appreciable amounts, has a greater molar absorbance than N-acetylsialic acid, so that previous determinations by other colorimetric methods are somewhat in error. E. J. H. BIRCH

3842. **A microchemical method for the detection and determination of shikimic acid.** M. K. Gaitonde and M. W. Gordon (Biochem. Lab., Inst. of Living, Hartford, Conn., U.S.A.). *J. Biol. Chem.*, 1958, **230** (2), 1043–1050.—The detection and determination depend on the oxidation of shikimic acid with HIO_4, and treatment of the product with aq. NaOH to give an intense yellow coloration, the extinction of which is determined at 380 mμ. The method is not directly applicable to tissue extracts that contain interfering substances. These are removed by paper chromatography; the acid can then be extracted from the paper and determined. Only shikimic acid, quinic acid and tryptophan give a yellow colour under the specified conditions. When quinic acid is also present, shikimic acid is determined first by measuring the extinction immediately after the addition of NaOH and glycine (to stabilise the colour) before a significant colour is produced by the quinic acid. J. N. ASHLEY

3843. **Fluorimetric determination of kynurenic acid and xanthurenic acid in human urine.** K. Satoh and J. M. Price (Cancer Res. Hosp., Med. Sch., Univ. of Wisconsin, Md., U.S.A.). *J. Biol. Chem.*, 1958, **230** (2), 781–789.—The two acids are adsorbed on Dowex-50 resin (H form) and are eluted simultaneously by washing with water. The kynurenic acid is rendered fluorescent by conc. H_2SO_4 and is then determined by a standard method. The xanthurenic acid is determined similarly in the presence of conc. alkali. The recoveries of both acids are 90 to 105%. Six samples per day may be analysed in duplicate for the two acids. Other quinoline derivatives do not interfere. J. N. ASHLEY

3844. **Screening test for steatorrhoea using iodine-131-labelled triolein.** M. Lubran and J. D. Pearson (West Middx. Hosp., Isleworth, England). *J. Clin. Path.*, 1958, **11** (2), 165–169.—A method of iodinating triolein with ^{131}I is described. Administration of 5 to 10 μC of this fat followed by measurement of radioactivity in a 3-day collection of stools presents an alternative test to the usual fat-balance estimation.

H. F. W. KIRKPATRICK

3845. **Determination of phenylpyruvate and phenylalanine in body fluids.** M. Petrovački (Inst. Clin. Chem., Fac. Pharm., Zagreb). *Acta Pharm. Jugoslav.*, 1957, **7**, 169–173.—The analytical methods for phenylpyruvate (**I**) and phenylalanine (**II**) have been adapted to the determination of these substances in phenylketonuric urine, serum, and cerebrospinal fluid. **II** can be determined in urine that has previously been freed of **I** and phenyl lactate by ether extraction at low pH. For standardisation, soln. of the pure compounds in normal urine were prepared. Good proportionality between optical density and concn. was found for both **I** and **II**. CHEM. ABSTR.

3846. **Adenosine mono-, di- and tri-phosphate, pyruvic kinase, hexokinase and polynucleotide phosphorylase assay.** S. Grisolia, L. C. Mokrasch and V. D. Hospelhorn (Kansas Univ. Med. Center, Kansas City, U.S.A.). *Biochim. Biophys. Acta*, 1958, **28** (2), 350–354.—The method described is based on the enzymic interconversion of the adenosine compounds and reaction of adenosine diphosphate with phosphoenol pyruvic acid to produce pyruvate, which is then determined by the method of Kachmar and Boyer (*J. Biol. Chem.*, 1953, **200**, 669). In each case, 0·03 μmole of the compounds and enzymes can be determined with reasonable specificity by different modifications of the method. The limitations of the methods are discussed. W. H. C. SHAW

3847. **Identification and chemical assay of epinephrine [adrenaline] in brain and other tissues.** P. A. Shore and J. S. Olin (Lab. of Chem. Pharmacol., Nat. Heart Inst., Bethesda, Md., U.S.A.). *J. Pharmacol.*, 1958, **122**, 295–301.—Noradrenaline and adrenaline in 0·01 *N* HCl are poorly extracted into butanol but the determination is facilitated by saturation of the aq. phase with NaCl. *Procedure*—Homogenise brain in 2 vol. of 0·01 *N* HCl, and transfer 6-ml samples of the homogenate to a 125-ml glass-stoppered bottle containing 7 to 8 g of solid NaCl and 60 ml of butanol. Shake for 1 hr., centrifuge, and transfer a 40-ml aliquot of the butanol extract to another 125-ml bottle containing 5 ml of 0·01 *N* HCl and 70 ml of heptane.

Shake for 5 min. then centrifuge. Transfer a 3-ml sample of the extract, containing 0·02 to 0·03 μg per ml, to a test-tube, and add 0·1 ml of buffer soln. (pH 5) followed by 0·1 ml of an iodine reagent. After 6 min. destroy the excess of iodine by adding 0·2 ml of $Na_2S_2O_3$ soln., then add 1 ml of alkaline ascorbate soln. Set aside for 45 min. and then activate the soln. at 400 mμ and read the resulting fluorescence at 520 mμ in a spectrophotometer. The method is simple and rapid and can be applied to the measurement of catecholamines in brain and other tissues. B. P. BLOCK

3848. **A method for estimating histamine in plasma.** H. M. Adam, D. C. Hardwick and K. E. V. Spencer (Dept. of Pharmacol., Univ. of Edinburgh). *Brit. J. Pharmacol.*, 1957, **12**, 397–405.—A chromatographic method of extracting histamine from the plasma and assaying the histamine on superfused guinea-pig ileum is presented. The plasma proteins are initially pptd. with trichloroacetic acid and centrifuged. Aliquots of the centrifugate are prepared for adsorption on buffered columns of Amberlite XE-64 resin at pH 8 and the histamine and other bases are eluted with HCl. The eluate is converted into the soln. for bioassay by the method of Adam *et al.* (*Brit. J. Pharmacol.*, 1954, **9**, 360). As little as 1 mμg per ml was recovered from the plasma of man and cats, and in recovery experiments from a concn. of 2 mμg of histamine per ml the mean recovery was 102 ± 15%. The determination requires two days to perform. B. P. BLOCK

3849. **Establishment of a colorimetric method of determination of histamine.** J. Thouvenot, N. Flavian and R. Weber (Fac. de Méd. de Paris, France). *Bull. Soc. Chim. Biol.*, 1957, **39** (12), 1511–1519.—The method of Rosenthal and Tabor (*J. Pharmacol.*, 1948, **92**, 425) has been studied with the specific object of rendering it sufficiently exact for use in following the distribution of histamine in human serum during dialysis. The protein soln. (2 ml) containing histamine (20 to 50 μg per ml) is deproteinised with 2 ml of 20% trichloroacetic acid (**I**), and filtered through fritted glass. The ppt. is washed twice with 1·8 ml of 10% **I** and the combined filtrate and washings are made up to 10 ml with **I** and placed in an ice bath. To the soln. are added, successively, 1 ml of diazo reagent (10 ml of 0·1% *p*-nitroaniline in 0·1 *N* HCl and 1 ml of 4% $NaNO_2$), 1·25 ml followed by 0·5 ml of 20% Na_2CO_3, and 0·3 ml of 5 *N* NaOH, shaking and replacing the mixture in the ice bath after each addition. The rose-coloured compound is extracted by shaking with 5 ml of *iso*butyl methyl ketone, which is allowed to separate in the ice bath, and the solvent layer is transferred to a tube containing 6 ml of a specified barbitone buffer (pH 7·7) to intensify the histamine colour and reduce the colour due to interfering substances. The extinction is read within 30 min. at 525 mμ. The details of the method must be closely adhered to, and the volumes of reagents, etc., measured precisely by means of micro-burettes. Under the given conditions, histamine can be measured in protein media with adequate accuracy only if its concn. lies between 20 and 50 μg per ml, the absolute error not exceeding 2 μg per ml. R. E. ESSERY

3850. **A simplified method for measuring serotonin [5-hydroxytryptamine] in tissues: simultaneous assay of both serotonin and histamine.** H. Weissbach, T. P. Waalkes and S. Udenfriend (Lab. of Clin. Biochem., Nat. Heart Inst., Bethesda, Md., U.S.A.). *J. Biol. Chem.*, 1958, **230** (2), 865–871.—The method depends on the pptn. of protein in the tissue extract with $Zn(OH)_2$, followed by direct spectrophotofluorimetric determination of 5-hydroxytryptamine in the filtrate by the method of Udenfriend *et al.* (*Science*, 1955, **122**, 972). 5-Hydroxytryptamine and histamine may be determined in the same tissue extract; after pptn. of the protein, both amines are extracted by butanol from the alkaline mixture. The butanol is passed through a cotton - acid succinate column, which separates the histamine from the 5-hydroxytryptamine. Each is eluted and the histamine is determined by the 1-fluoro-2:4-dinitrobenzene method. The method is applicable to blood, plasma and tissues. J. N. ASHLEY

3851. **Determination of total nucleic acids by ultra-violet absorption methods.** J. M. Webb (Nat. Inst. of Allergy and Infectious Diseases, Bethesda, Md., U.S.A.). *J. Biol. Chem.*, 1958, **230** (2), 1023–1030.—In the presence of an excess of trichloroacetic acid (added after hydrolysis) there is no significant change in the wavelength of max. absorption or extinction value in hydrolysates from tissues that have been hydrolysed for 15 or 60 min. The max. for tissue hydrolysates is ≃ 265 mμ, which agrees well with that of a soln. of deoxyribonucleic acid and ribonucleic acid (1:1), and of a synthetic mixture, based on the tetranucleotide formula, of components that would arise from the hydrolysis of the nucleic acids. A correction is obtained for the ultra-violet absorption of trichloroacetic acid at wavelengths as low as 265 mμ by treating the control soln. similarly to tissue hydrolysates with respect to heating, addition of extra trichloroacetic acid, and dilution. J. N. ASHLEY

3852. **Separation of nucleotides by continuous ionophoresis.** R. Bergkvist (Inst. of Biochem., Univ. of Lund, Sweden). *Acta Chem. Scand.*, 1958, **12** (3), 555–560.—The method previously described (*cf. Anal. Abstr.*, 1958, **5**, 2331) has been modified so that the mixture to be separated is fed continuously into the apparatus, and the separated components can be continuously removed. Acetate buffer at pH 4·15 (ionic strength 0·033) travels down a Whatman No. 1 paper curtain and drips from tongues cut in the bottom edge while a potential difference of 320 V is applied by platinum wires across the sheet. A steady state is reached after 6 hr. with a current of 17 mA, and the mixture of nucleotides is then applied regularly from a motor-driven syringe to a suitable spot on the paper. Nearly all the purine and pyrimidine nucleotides could be separated into four groups by using a preliminary paper chromatography (*cf. loc. cit.*) and these groups were eluted and concentrated on Norit before being separated into components by the continuous electrophoresis. Recoveries of 85 to 95% were obtained. Only diphosphopyridine nucleotide was slightly decomposed during separation. Analysis and identification were effected by means of the optical absorption at 250, 260 and 270 mμ. E. J. H. BIRCH

3853. **The effect of the width of paper on R_F values of amino acids.** C. R. Willms and W. M. Harding (State Teachers' Coll., San Marcos, Texas, U.S.A.). *Mikrochim. Acta*, 1958, (3), 411–414 (in English).—The R_F values of typical amino acids (with high, low, and intermediate isoelectric points)

are increased by 25 to 40% or more if the rectangular paper strip is very narrow (2 to 4 mm) or the sample is placed ≃ 5 mm from the edge of a wide strip. The effect is obtained with a basic as well as an acid solvent. W. J. BAKER

3854. **Sensitivity of the ninhydrin test in chromatographic analysis.** J. Opieńska-Blauth, H. Kowalska and M. Pietrusiewicz. *Ann. Univ. M. Curie-Skłodowska, D*, 1957, **11**, 175–198.—The ninhydrin test is more sensitive than the isatin test for most amino acids except proline. The sensitivity of the ninhydrin test is a direct function of the concn. of ninhydrin in the developer. Soln. more concentrated than 0·5% should not be used because of paper staining. Amino acids which have the highest R_F values show the greatest loss of sensitivity during chromatographic partition in phenol. Methionine shows a high sensitivity before, and a low one after, chromatographic partition, and the sensitivity of histidine changes little during partition. Most amino acids show a higher sensitivity at room temp. as compared to the ninhydrin reaction carried out at 100°. CHEM. ABSTR.

3855. **Identification of certain amino acids and derivatives by paper chromatography with the use of the system ethyl acetate - pyridine - water.** J. Moreno Calvo and A. Santos Ruiz (Inst. Espagnol de Physiol. et Biochim., Centre Exp. du Froid, Serrano 150, Madrid, Spain). *Bull. Soc. Chim. Biol.*, 1957, **39** (12), 1557–1564.—The R_F values are given for several amino acids and their derivatives, obtained by the use of the non-aq. layer of the system ethyl acetate - pyridine - water (2:1:2), previously employed for the separation of carbohydrates (*cf.* Jermyn and Isherwood, *Brit. Abstr. C*, 1949, 332). The acids were applied to Whatman No. 1 paper in amounts of 3 μl of fresh 1% soln., the paper was then rolled into a cylinder and stood upright with 1 cm of its edge immersed in the solvent contained in a Petri dish on a glass plate, the whole being then covered with a bell-jar, sealed with stopcock grease, and development was allowed to proceed for 21 hr. Spots were revealed by spraying the paper with 0·2% ninhydrin in a freshly prepared mixture of *n*-butanol - 2 *N* acetic acid (95:5). The acids can be applied to the paper as salts, the pyridine serving as a weak base for neutralisation; optical isomerism has no influence on R_F values. Some amino acids which are not shown by the more usual reagents are revealed by this procedure. R. E. ESSERY

3856. **Paper chromatography of amino acids and peptides with ethanol.** D. Lebez and A. Dekleva-Likar ("J. Stefan" Inst., Ljubljana, Yugoslavia). *Rep. "J. Stefan" Inst., Ljubljana*, 1957, **4**, 181–184 (in English).—Aq. ethanol (75%) is a suitable solvent for the separation of amino acids and peptides. Proteins do not migrate with this solvent and consequently do not interfere. The R_F values of 32 peptides and amino acids in 75% aq. ethanol are listed in comparison with the corresponding values in other ethanolic solvent mixtures. G. BURGER

3857. **A new colorimetric procedure for the determination of lysine.** J. C. Sanahuja and D. Seoane (Dept. of Food Science, Fac. of Pharm., Univ. of Buenos Aires). *An. Bromatologia*, 1958, **10** (1), 165–173.—The method is based on the coupling of lysine with diazotised *p*-nitroaniline and spectrophotometric measurement of the coloured complex produced. *Procedure*—To 5 ml of sample containing from 20 to 300 μg of lysine add 1 ml of diazotised *p*-nitroaniline [25 ml of 0·3% *p*-nitroaniline soln. (in aq. HCl) with 0·75 ml of 10% $NaNO_2$ soln.]. After 5 min. add 3 ml of 16% Na_2CO_3 soln. and mix. After a further 20 min., dilute to 15 ml with solvent (40 g of NaOH, 100 ml of 95% ethanol and 140 ml of water) and read the extinction at 520 mμ. In pure soln. the error is ± 2%. Other basic amino acids interfere. E. C. APLING

3858. **Quantitative determination of *p*-hydroxyanilinoacetic acid.** G. P. Faerman and Ya. M. Veprik. *Zhur. Nauch. i Prikl. Fotogr. i Kinematogr.*, 1957, **2** (2), 110–115; *Ref. Zhur., Khim.*, 1958, Abstr. No. 997.—*p*-Hydroxyanilinoacetic acid (*p*-hydroxy-N-phenylglycine) (**I**) is determined by potentiometric titration with $Ce(SO_4)_2$ in an atmosphere of N at 25°. The first change of potential occurs at the equivalence point corresponding to the loss by a molecule of **I** of two electrons, and the second at the equivalence point corresponding to the loss by a molecule of **I** of four electrons. With a concn. of 0·0006 *M*, the accuracy of the titration is ± 0·7%. C. D. KOPKIN

3859. **Use of a non-ionic detergent in paper electrophoresis of serum proteins.** J. J. Downs, E. Geller, K. D. Lunan and L. T. Mann (U.S. Army Med. Lab., 90 Church St., New York, U.S.A.). *J. Lab. Clin. Med.*, 1958, **51** (2), 317–320.—Improved results are obtained in conventional paper-electrophoresis methods by the addition of 0·2% of Sterox SE to the electrophoresis buffers. Results for albumin show good agreement with those obtained by salt-fractionation procedures or moving-boundary electrophoresis. Values for serum proteins from 49 normal subjects are given. W. H. C. SHAW

3860. **A review of several colour and fluorescence reactions of steroids.** O. Siblíková and I. M. Hais (Res. Inst. Pharm. and Biochem., Prague). *Československ. Farm.*, 1958, **7** (1), 1–13.—Reactions on paper have been tested for 85 compounds, and colour shades and relative intensities of the spot reactions are tabulated for the following reagents—aq. iodine soln., H_3PO_4, conc. H_2SO_4, soln. of $SbCl_3$ in $CHCl_3$, trichloroacetic acid in $CHCl_3$, Dragendorff's reagent, 2:4-dinitrophenylhydrazine soln., Zimmermann's reagent, 10% NaOH soln., *p*-phenylenediamine phthalate, blue tetrazolium, and Liebermann's reagent. The reactions are divided into three groups—(*a*) less specific reactions ($SbCl_3$, H_3PO_4, H_2SO_4); (*b*) group-specific (selective) reactions (blue tetrazolium, Zimmermann's reagent, etc.); (*c*) reactions characteristic only for a small group of compounds (Liebermann's reaction for Δ^5-3β-hydroxysterols, etc.). J. VOLKE

3861. **The influence of autoxidation on the chemical assay of cholesterol.** L. N. Norcia (Univ. Oklahoma Sch. Med., Oklahoma City, U.S.A.). *J. Amer. Oil Chem. Soc.*, 1958, **35** (1), 25–27.—Cholesterol, purified by its conversion into the dibromide, and two commercial samples showed no spectral differences when compared by i.r. examination in CS_2 soln. and by the potassium bromide disc technique. All three samples were then assayed by the Schoenheimer and Sperry method, and each gave a specific extinction coeff. of 4·35 (625 mμ). Similar samples, when autoxidised by air in an aq. colloidal suspension, stabilised with Na stearate, for 3·5 to 4 hr. at pH 8·2, showed losses in the

cholesterol content by an amount approx. equal to the percentage conversion of cholesterol into diols and ketones. Assays of the same materials by the Liebermann - Burchard method showed no corresponding loss on aerial oxidation. Cholesterol is not oxidised by refluxing in an ethanol - diethyl ether mixture (3:1) in the presence of maize oil.

G. R. WHALLEY

3862. **Separation of oestrogens by paper chromatography.** H. Struck (Forschungslab. der Chem.-Pharm. Fabrik Adolf Klinge, G.m.b.H., München, Germany). *Naturwissenschaften*, 1958, **45** (2), 41–42.—The oestrogens are spotted on acetylated cellulose (S. & S. 2043b) and developed with 80% aq. methanol for 5 hr. After drying at room temp., the chromatograms are passed through a soln. of 10% $SbCl_5$ in CCl_4 and dried for 3 min. at 80°. Characteristic colours are obtained for the different oestrogens (oestrone, brown; oestradiol, yellow - brown; oestriol, red; equilin, grey - brown; equilenin, green - brown). After a time all these colours change to a dirty brown. E. KAWERAU

3863. **Isolation and estimation of "free" oestrogens in human placentae.** E. Diczfalusy and P. Lindvist. *Acta Endocr., Copenhagen*, 1956, **22**, 203–223.—The method described is applicable to minute amounts of oestrone, 17β-oestradiol and oestriol in small samples of tissues. It has been developed from the urinary oestrogen method of Brown, and involves solvent partition, chromatographic separation of oestrogen methyl ethers on alumina columns and an improved Kober colour reaction. G. F. SOMERS

3864. **Determination of urinary pregnanetriol.** W. Herrmann and L. Silverman (Yale Univ. School of Med., New Haven, Conn.). *Proc. Soc. Exp. Biol. Med.*, 1957, **94**, 426.—Aliquots (50 ml) of urine are acidified with acetic acid, incubated with glucuronidase for 24 hr. and extracted with ether. The ether extracts are washed with *N* NaOH, dried under nitrogen and transferred with benzene to a Florisil column. 17-Oxosteroids and pregnanediol are eluted with 2% ethanol in benzene and pregnanetriol with 4% ethanol in benzene. The pregnanetriol extract is dried, taken up in acetic acid and oxidised with sodium bismuthate. Excess of bismuthate is removed with sodium metabisulphite, and the oxosteroid is extracted with ether and determined by the micro-method of Holtouff and Koch, with *m*-dinitrobenzene in KOH, comparison being made with dehydro*epi*androsterone as standard. Recoveries of $\simeq$ 78·5% were obtained.

G. F. SOMERS

3865. **Hydrolysis, extraction and purification of metabolic oestrogenic products present in urine from cows.** E. Rota (Turin Univ., Italy). *Ric. Sci.*, 1958, **28** (2), 362–376.—A method for the detection of oestrogens involves separating them from interfering substances by partial reduction of the phenoxides, transformation to non-alkali-soluble benzoates, and successive separation and purification treatments with acetic acid, ethanolic NaOH and aq. $MgCl_2$. The purified extract is treated in ethanolic NaOH soln. with a few drops of HCl and diazo reagent (Ehrlich). No colour should appear at this stage but, on addition of aq. NH_3, a red - orange colour should develop which may be used as a basis for an approx. quant. estimation.

L. A. O'NEILL

3866. **Determination of 17-oxosteroids in urine.** W. Staib and W. Schild (Physiol.-Chem. Inst., Med. Akad., Düsseldorf, Germany). *Klin. Wochschr.*, 1958, **36** (4), 166–169.—An apparatus is described for the fractional elution of 17-oxosteroids from an alumina column with benzene - ethanol. In a complete run of 70 to 80 fractions, 8 to 9 peaks were observed, three of which were identified by infra-red analysis to be dehydro*epi*androsterone, androsterone and aetiocholanolone.

H. F. W. KIRKPATRICK

3867. **Analysis of neutral urinary 17-oxosteroids. Selective hydrolyses: identification and determination by paper chromatography.** E. E. Baulieu, S. H. Weinmann and M. F. Jayle (Lab. de Chim. Biol., Fac. de Méd., 45, rue des Saints-Pères, Paris). *Bull. Soc. Chim. Biol.*, 1957, **39** (12), 1371–1394.—A complex procedure, derived from existing techniques, has been developed. It comprises the extraction of total ketosteroids from urine with ether - ethanol, hydrolysis of the sulphates with dioxan - trichloroacetic acid and of the glucuronides with β-glucuronidase from *Helix pomatia*, and chemical purification by treatment with Girard reagent T, separation on a column of Florisil and pptn. with digitonin. The three groups so obtained are then separated by paper chromatography, the 11-deoxy-17-oxosteroids with light petroleum - propane-1:2-diol, the 3β-hydroxy-17-oxosteroids with methyl*cyclo*hexane - propane-1:2-diol, and the 11-hydroxy-17-oxosteroids with hexane - benzene - propane-1:2-diol. The spots are revealed by dipping the paper successively in 10% ethanolic KOH and 2% ethanolic *m*-dinitrobenzene. The method is made semi-quant. (precision $\pm$ 20%) by comparison with reference spots of pure steroids. Colours are also given with 15% aq. H_3PO_4 and with conc. H_2SO_4 - ethyl acetate (5:1). The stages of the method are fully discussed, and the procedure is compared with those previously advanced.

R. E. ESSERY

3868. **Per-extraction. A procedure to extract C_{21} steroids from tissues and blood.** F. DeVenuto, S. J. Mulé and U. Westphal (U.S. Army Med. Res. Lab., Fort Knox, Kentucky, U.S.A.). *Arch. Biochem. Biophys.*, 1958, **73** (2), 451–465.—The procedure described is suitable for the extraction and purification of steroids from biological materials for subsequent paper chromatography or other purposes. After homogenisation in the presence of diluted ethanol, the sample is transferred to a Cellophane bag which is placed in the extraction chamber of a Soxhlet apparatus. Continuous extraction is then carried out with $CHCl_3$ or other suitable solvent. Recovery of steroid is complete with cortisol, but declines with decreasing polarity of the steroid. The modifications to the general method necessary for quant. recovery of corticosterone, deoxycorticosterone and progesterone are described.

W. H. C. SHAW

3869. **Problems related to the chemical assay of corticosteroids in human body fluids.** R. Borth (Clinique Univ. de Gynécologie et d'Obstetrique, Geneva, Switzerland). *Acta Endocr., Copenhagen*, 1956, **22**, 125–135.—A rapid method for clinical use for the determination of 17:21-dihydroxy-20-oxosteroids is based on the application of the reaction of Porter and Silber to a crude *n*-butanol extract, but the method is only approximate. Reliable determinations of total 17:20-dihydroxy- and 17-hydroxy-20-oxosteroids are made by the

Zimmermann reaction after converting them into 17-oxosteroids with sodium bismuthate. If the sole interest is in checking whether the corticosteroid levels increase after the administration of corticosteroids or corticotrophin, any convenient method will do, but for more fundamental investigations paper-chromatographic methods are recommended. G. F. SOMERS

3870. **Rapid and sensitive method for the determination of chymotrypsin and trypsin activity.** C. J. Martin, J. Golubow and A. E. Axelrod (Pittsburgh Univ. Med. Sch., Pa., U.S.A.). *Biochim. Biophys. Acta*, 1958, **27** (2), 430–431.—The method is based on the use of N-benzyloxycarbonyl-L-tyrosine *p*-nitrophenyl ester as substrate. Enzyme activity is estimated from the increase in extinction at 400 mμ due to liberation of nitrophenoxide ion at pH 8. Reaction rates may be measured with 3 mμg of chymotrypsin and 7 mμg of trypsin per ml of solution. W. H. C. SHAW

3871. **Micro-determination of ribonuclease.** J. C. Houck (Georgetown Univ. Hosp., Washington, D.C., U.S.A.). *Arch. Biochem. Biophys.*, 1958, **73** (2), 384–390.—The method described is based on the formation of turbidity, which can be measured at 400 mμ, when a solution of ribonucleic acid at 37° is mixed with a solution containing serum albumin and the pH is adjusted to 4·0. The turbidity, stabilised with gelatin, decreases with ribonuclease activity; a satisfactory calibration curve is obtained with 1 to 50 mμg of crystalline ribonuclease. The method is not affected by the relative purity of either the enzyme or the substrate. W. H. C. SHAW

3872. **Colorimetric determination of urinary lipase.** M. M. Nachlas and R. Blackburn (Dept. of Surgery, Sinai Hosp. of Baltimore, Md., U.S.A.). *J. Biol. Chem.*, 1958, **230** (2), 1051–1061.—Urinary lipase is determined with the use of a new chromogenic substrate, 2-naphthyl caprylate. The liberated 2-naphthol is coupled with tetrazotised *o*-dianisidine, and the colour density of the resulting azo dye is measured colorimetrically. Urine contains an antilipase which can be separated from the enzyme only by dialysis. Use of the new substrate avoids dialysis of each urine sample because under the specified conditions hydrolysis of the ester occurs much more rapidly than does inhibition of the enzyme. J. N. ASHLEY

See also Abstracts—**3614**, Detection of Cu in biological materials. **3785**, Determination of proline. **3795**, Determination of purines and pyrimidines. **3939**, Automatic pipetting attachment for Van Slyke manometric apparatus for determining CO_2. **3943**, Apparatus for removal of salts from amino-acid soln. **3945**, Micro-extractor for biological materials.

Drugs

3873. **Systematic procedure for the identification of dangerous drugs, poisons and narcotics by ultraviolet spectrophotometry.** L. W. Bradford and J. W. Brackett (Lab. Crim., Santa Clara Court House, San José, Calif., U.S.A.). *Mikrochim. Acta*, 1958, (3), 353–382 (in English).—A systematic scheme for the separation and identification of the relatively pure substances is given. The compounds in the various fractions obtained by the extraction described can be identified spectrophotometrically (in specified solvents) by referring the data to the four tables provided. These list the absorptivities and absorption maxima, between 220 and 400 mμ, of 166 organic compounds, 25 vegetable materials and 4 human biological specimens at given concn. Compounds that do not show specific absorption, and those that can be identified only by change in u.v. spectra on alteration of pH are listed separately. For the spectrophotometric measurements, matched cells are used, and the dilutions should ensure absorbance readings from 0·1 to 1·0 at max. extinction. The concn., in the sample, of any substance that has been identified can be determined approx. from the available data. W. J. BAKER

3874. **Spectrophotometric determination of alkaloids in the ultra-violet range at 2537 A.** H. Thies and C. H. Sorgenfrey (Inst. für Pharm. u. Lebensmittelchemie, München Univ.). *Arch. Pharm., Berlin*, 1958, **291**, 68–76.—Results of experiments with several alkaloids indicate that extinction measurements at 2537 A are very accurate and reproducible, and can serve as an optical parameter for characterisation of organic substances. They can also be applied to the quantitative determination of small amounts of alkaloids, the accuracy lying within the usual limits of determination by other methods. The temp. coeff. of extinction is different for different alkaloids and its values are given for several substances. (29 references.) M. H. SAWISTOWSKA

3875. **The application of bromoauric acid to the microchemical identification of alkaloids and organic bases.** G. Sandri (Ist. di Chim. Agraria, Univ. Bologna, Italy). *Mikrochim. Acta*, 1958, (2), 248–252.—The products obtained from pyridine and quinoline bases with bromoauric acid are described. The crystalline ppt. given by caffeine, theobromine and theophylline are of special interest since they permit a differentiation of these bases. D. F. PHILLIPS

3876. **Potentiometric titration of certain salts of alkaloids and synthetic bases.** Ya. M. Perel'man. *Sb. Nauch. Trud. Leningr. Khim.-Farmatsevt. Inst.*, 1957, **2**, 38–55; *Ref. Zhur., Khim.*, 1958, Abstr. No. 1001.—Based on examples of the titration of the hydrochlorides of morphine (**I**), codeine (**II**), salsoline (**III**), salsolidine (**IV**), procaine (**V**), ephedrine (**VI**) and Dimedrol (**VII**), hyoscine hydrobromide (**VIII**) and amphetamine sulphate (Phenamine) (**IX**), the possibility is indicated of using the glass electrode for direct replacement reactions. The **I**, **II**, **VII** and **VIII** are easily titrated in aq. soln. at concn. of 0·01 to 0·03 *M*; weaker soln. and salts of bases of $pK < 5$ are titrated in aq. ethanolic soln., **V** and **VI** at an ethanol concn. of 80 to 85%, and **III**, **IV** and **IX** at an ethanol concn. of 95 to 96%. C. D. KOPKIN

3877. **Polarographic determination of papaveraldine.** J. Křepinský (State Inst. for Control of Drugs, Prague). *Československ. Farm.*, 1958, **7** (1), 13–16.—In 10% acetic acid papaveraldine gives a two-electron wave corresponding to the reduction of the carbonyl group ($E_{\frac{1}{2}} = -0{\cdot}36$ V *vs.* the S.C.E.). The dependence of the wave height on the concn. (0·002 to 0·01%) is linear. Papaverine and papaverinol do not interfere. The degree of decomposition of papaverine injections was studied by this method. J. VOLKE

3878. **Titrations in non-aqueous media in pharmaceutical analysis. III. Determination of arecoline-acetarsol.** L. Šafařík (State Inst. for Control of Drugs, Prague). *Československ. Farm.*, 1958, **7** (1), 16–17.—A titration in glacial acetic acid with 0·05 *N* $HClO_4$ was developed, with potentiometric or visual control (crystal violet). Both substance and tablets may be analysed by this method.
J. VOLKE

3879. **Colorimetric determination of nitrogenous substances in complex mixtures as reineckates.** Mlle Masse (Lab. Nat. de Contrôle des Médicaments, Paris). *Pharm. Acta Helv.*, 1958, **33** (2), 80–84.—To determine betaine in the presence of hexamine, heat the soln. of pH 1, add a saturated aq. soln. of ammonium reineckate, cool in ice for 30 min., filter through sintered glass (G4), dissolve the ppt. in acetone and measure at 525 mμ. To determine piperazine in the presence of hexamine, heat the soln. of pH 2 to 4, add a saturated soln. of ammonium reineckate, cool in ice for 90 min., collect the ppt., dissolve and measure as above. To determine Strychnal (ethylbetaine sulphate of strychnic acid) in the presence of thiamine, add Na_2CO_3 soln. and a saturated soln. of ammonium reineckate, cool in ice for 30 min., collect the ppt., dissolve and measure as above; thiamine may be determined in the filtrate.
A. R. ROGERS

3880. **Determination of purine derivatives in non-aqueous solution. II. Potentiometric and visual titration of theobromine and of diprophylline (dihydroxypropyltheophylline). Differential titration of the constituents of Diuretin (theobromine and sodium salicylate).** B. Salvesen (Univ. Farm. Inst., Oslo, Norway). *Medd. Norsk Farm. Selsk.*, 1958, **20** (2), 21–25.—Theobromine and diprophylline are titrated in the presence of acetic anhydride according to the method devised for the titration of caffeine (*Anal. Abstr.*, 1958, **5**, 3115). *Procedure*—Dissolve 0·3 to 0·4 g of theobromine or diprophylline in 15 ml of acetic anhydride by heating to boiling, with addition of 5 ml of glacial acetic acid for theobromine. After cooling, add 20 ml of benzene and 5 drops of 1% methyl violet. Titrate with 0·1 *N* $HClO_4$ in glacial acetic acid to a colour change from greenish yellow to green. For theobromine and Na salicylate dissolve 0·35 g in 5 ml of glacial acetic acid and 20 ml of acetic anhydride and heat to boiling. After cooling add 20 ml of benzene and 3 drops of Tropaeolin OO and titrate with 0·1 *N* $HClO_4$, from yellow to a clear orange (V_a ml); then add 7 drops of crystal violet and titrate the theobromine content to reddish yellow (= V_b ml) (1 ml of 0·1 *N* $HClO_4$ ≡ 0·01601 g of Na salicylate and 0·01802 g of theobromine or 0·02022 g of anhydrous Na theobromine). The theobromine content is given by the vol. (V_b ml) used for the second titration; the Na salicylate is given by the difference, $V_a - V_b$. The indicator error is not more than 0·1%.
P. HAAS

3881. **Determination of santonin in *Artemisia maritima* L., subspec. *monogyna* Gams, growing in Slovakia.** G. Dušinský and M. Tyllová (State Inst. for Control of Drugs, Bratislava, Czechoslovakia). *Chem. Zvesti*, 1958, **12** (2), 102–107.—Benzene extraction, followed by paper chromatography and polarography, is used. *Procedure*—Moisten the dried pulverised sample (5 g) with aq. NH_3 (10%) (4 ml) and extract in a Soxhlet apparatus with benzene (90 ml) (12 hr.). Distil off the benzene, dry the residue with hot air and heat for 25 min. with $Ba(OH)_2$ soln. (5%) (55 ml) on a water bath (70°). Cool, filter, and wash the ppt. with H_2O, add HCl (10%) to the filtrate till acid (Congo red indicator) and heat for 25 min. (60° to 70°) on a water bath, cool and extract with 25 ml, 3 × 20 ml, 15 ml and 10 ml of $CHCl_3$. Evaporate the combined extracts to 10 ml. Place 0·02 to 0·1 ml (≡ 50 to 200 μg of santonin) on Whatman paper strips (2 cm × 30 cm) and as solvent use light petroleum - benzene - $CHCl_3$ (5:4:1) satd. with aq. NH_3 and diluted with 2% of methanol (2 ml). After 45 to 60 min. detect the santonin spots with sodium reagent (heat 12·7 g of Na with 87 g of methanol under reflux), cut out the red spots (2 cm × 2 cm) and elute for 20 min. with glacial acetic acid (1·5 ml). Add H_2O (10·5 ml), transfer the soln. to a polarographic vessel, remove O by passing N for 15 min. and register the wave from − 0·4 V. Make a calibration curve. Amounts of 0·2 to 0·3% of santonin in plant material can be determined.
J. ZÝKA

3882. **Colorimetric determination of rutin with quadrivalent titanium salts.** U. Bilińska (Inst. Inorg. Chem., Wrocław, Poland). *Acta Polon. Pharm.*, 1958, **15** (2), 123–128.—The concn. of rutin in an alcoholic extract is measured by the intensity of colour of an orange - yellow complex formed with $TiOSO_4$. The pH is adjusted to 5·8 with 3 *N* Na acetate. The colour which is stable for 0·5 hr. is measured by a photo-electric absorptiometer. The results are read from a graph obtained from readings of standard solutions. This method enables low concn. (17 p.p.m.) of rutin to be determined in pharmaceutical products as well as in original substances.
H. DMOWSKA

3883. **Polarographic determination of rutin in pharmaceutical preparations.** J. Davídek and O. Manoušek (Res. Inst. Food Technol., Prague). *Československ. Farm.*, 1958, **7** (2), 73–75.—The method is based on the determination of nitroso derivatives of rutin (and quercetin). The drug (0·1 g) is dissolved in methanol (25 ml), and the soln. (0·5 ml) is mixed with methanol (0·5 ml), 0·2 *N* H_2SO_4 (5 ml) and 3 *M* KNO_2 (2 ml) in a polarographic vessel. After bubbling the soln. with nitrogen for 2·5 min., 2·5 *M* Na acetate (2 ml) is added, and the bubbling is continued for a further 4 min. The polarographic curve is recorded, and the first step is measured. The height of the step is evaluated by means of a calibration curve or by standard addition. Even 10^{-6} *M* soln. of rutin may be analysed; the heights of the waves are independent of time. Ascorbic acid and other compounds likely to be present do not interfere. The error is ± 4%.
J. VOLKE

3884. ***Neurospora* plate method for testing antimetabolites.** R. Fuerst and W. M. Skellenger (Texas Univ., Houston, U.S.A.). *Antibiot. & Chemother.*, 1958, **8** (2), 76–80.—In the agar-plate method described, 40 μl of soln. containing 2·5 μg or more of the compound under test is placed in 5-mm-diameter holes punched in the agar. A filtered microconidial suspension of *Neurospora crassa* (Em 5256 A) (10 μl) is then added to each hole and the plate is incubated at 37° for 22 to 24 hr. Activity is determined from the amount of compound required to give 50 or 25% of the growth-zone diameter in comparison with controls. Results for 7 antimetabolites are given.
W. H. C. SHAW

3885. **Analysis of acetylsalicylic acid chloride.** L. M. Litvinenko, V. P. Polyakov, A. P. Grekov and A. M. Chernetskaya. *Med. Prom., SSSR*, 1957, (4), 42–43; *Ref. Zhur., Khim.*, 1958, Abstr. No. 4358.—The method consists in mixing a benzene soln. of acetylsalicylic acid chloride with a benzene soln. of aniline; the quantity of the latter is $\simeq 3$ times as great as that of the acid chloride (in molar ratios). Titrate the excess of aniline potentiometrically with $NaNO_2$ soln. with an indicating platinum electrode. The presence in the mixture of acetylsalicylanilide does not cause interference. The method has been tested on a series of acid chlorides; the error is $\not> 1\%$; the analysis takes $\not> 30$ min. C. D. KOPKIN

3886. **Examination of some intermediates of diethylstilbene-4:4′-diol [stilboestrol].** I. Gyenes and B. Stefkó (Anal. Lab., Pharm. Factory of Kőbánya, Budapest). *Magyar Kém. Foly.*, 1958, **64** (1), 16–17.—The following compounds were studied—*p*-hydroxypropiophenone (**I**), the *iso*propylidene ether of 3:4-di-(*p*-hydroxyphenyl)-hexane-3:4-diol (prep. described) and 4:4-di-(*p*-hydroxyphenyl)hexan-3-one. Compound **I** was titrated in anhyd. acetone or acetone - pyridine with 0·1 *N* K methoxide, with 0·1% azo violet in chlorobenzene as indicator. The other two compounds were titrated in neutralised anhyd. acetone - pyridine - methanol (70:30:2) with K methoxide, with azo violet as indicator. A. G. PETO

3887. **Determination of 8-hydroxyquinoline-5-sulphonic acid.** V. Velich (State Inst. for Control of Drugs, Prague). *Chem. Listy*, 1958, **52** (2), 346–348.—The sample is treated with iodine, the excess of which is titrated with $Na_2S_2O_3$ soln. *Procedure*—Dissolve the sample (2·2 to 2·25 g) in NaOH soln. (10%) (100 ml) and H_2O (50 ml) and dilute with H_2O to 250 ml. Measure 50 ml into a glass stoppered flask and add 50 ml of 0·1 *N* iodine. Set the mixture aside in the dark (5 min.) and add H_2SO_4 (25%, w/v) (25 ml) or HCl (11%, w/v) (30 ml) and KI (0·5 g). After 1 min. titrate with 0·1 *N* $Na_2S_2O_3$ (starch indicator). The error is $\pm 0{\cdot}2\%$. When determining 8-hydroxyquinoline in tablets with amidopyrine, the sample is dissolved in NaOH soln. (3%), the undissolved residue is filtered off, and the amidopyrine is extracted with $CHCl_3$. Suppositories can be dissolved in warm water. J. ZÝKA

3888. **Potentiometric titrations of drugs possessing a blocked aromatic amino group. II. Determination of some pharmaceutically important sulphones (Diaphenylsulfon, Sulfon, Rodilone) [dapsone, 4-carboxymethylamino-4′-aminodiphenyl sulphone, 4:4′-diacetylaminodiphenyl sulphone].** J. Blažek and Z. Stejskal (State Inst. for Control of Drugs, Prague). *Českosl. Farm.*, 1958, **7** (1), 23–25.—The test substance ($\simeq 500$ mg) is boiled under reflux with 12·5% HCl (100 ml) for 30 min. After cooling, the contents are transferred to a 250-ml flask and diluted to vol. with conc. HCl. An aliquot is mixed with fuming HCl and titrated with 0·1 *N* $NaNO_2$. A two-electrode system (platinum and graphite) is directly connected with the galvanometer. At the equiv. point, the deflection of the galvanometer is constant. The average error in determining the compounds mentioned above was $\pm 1\%$. J. VOLKE

3889. **Procedure for the isolation and determination of Trasentin (2-diethylaminoethyl diphenylacetate hydrochloride) [adiphenin hydrochloride] in solutions.** A. J. Taraszka and A. D. Marcus (Coll. of Pharm., Rutgers Univ., Newark, N.J.). *J. Amer. Pharm. Ass., Sci. Ed.*, 1958, **47** (2), 99–101.—Prepare a chromatographic column of silicic acid (20 g) with 0·5 *M* acetate buffer of pH 4·8 (20 ml) as internal phase. Mix an aq. soln. of the sample (containing < 250 mg of adiphenin hydrochloride) with an equal wt. of silicic acid; form into a slurry with CCl_4 and transfer quant. to the column. Elute with $CHCl_3$ at a rate of $\not> 2$ ml in 3 min. and collect 100 ml of eluate. To a 10-ml aliquot add 0·1% 1-naphtholbenzein soln. (5 drops) and titrate with 0·01 *N* $HClO_4$ in glacial acetic acid. Neither 2-diethylaminoethanol (which remains on the column) nor diphenylacetic acid interferes with the determination. The recoveries in 12 experiments ranged from 99·1 to 100·7%. A. R. ROGERS

3890. **Titrations in non-aqueous media in pharmaceutical analysis. IV. Determination of D-4-amino-3-isoxazolidone (cycloserine).** L. Šafařík and V. Špinková (State Inst. for Control of Drugs, Prague). *Českosl. Farm.*, 1958, **7** (2), 76–77.—The sample (50 to 60 mg) is dissolved in glacial acetic acid, 2 drops of a soln. of crystal violet are added, and the soln. is titrated with 0·05 *N* $HClO_4$. Both visual and potentiometric control (platinum and graphite electrodes in a standard soln. of Cu acetate in glacial acetic acid) can be used, even with tablets and capsules. In contrast with the polarimetric and Kjeldahl methods, lactose and isoniazid do not interfere. J. VOLKE

3891. **Quantitative analysis of an anti-neuralgic mixture containing phenylsemicarbazide.** C. Stainier and C. Lapière (Inst. de Pharm., Univ. Liége, Belgium). *Pharm. Acta Helv.*, 1958, **33** (2), 85–94.—Directions are given for the determination of phenylsemicarbazide (**I**), amidopyrine (**II**), phenazone (**III**), caffeine (**IV**) and phenacetin (**V**) in admixture. Recoveries range from 96·9 to 103·7%. *Procedure*—Extract a portion of the sample with CS_2, dissolve the residue in ethanol and determine **I** iodimetrically by the method of Doucet (*cf. J. Pharm. Chim.*, 1923, **27**, 361). Evaporate the CS_2 extract to dryness under reduced pressure and determine **II** iodimetrically by the method of Schulek and Menyhardt (*cf. Z. anal. Chem.*, 1932, **89**, 426). Extract another portion of the sample with CS_2, evaporate, precipitate **II** with cadmium thiocyanate and filter; extract the alkaline filtrate with dichloromethane and determine **III** in the extract iodimetrically by the method of Kolthoff (*cf. Pharm. Weekbl.*, 1923, 194). To determine **IV**, extract a portion of the sample with CCl_4, evaporate under reduced pressure and remove **II** and **III** by pptn. with Mayer's reagent by the method of Domange and Pinguet (*cf. Ann. Pharm. Franç.*, 1951, **11**, 651); extract **IV** from the filtrate with $CHCl_3$, evaporate, dry and weigh. To determine **V**, extract a portion of the sample with a saturated aq. soln. of **V**, dry the residue, extract with ether, dry and weigh (*cf.* Domange and Pinguet). A. R. ROGERS

3892. **Complexometric titrations in pharmaceutical analysis. XVII. Determination of bismuth.** F. Buben and J. Körbl (Res. Inst. Pharm. and Biochem., Prague). *Českosl. Farm.*, 1958, **7** (2), 78–79.—Bismuth ions are titrated at pH $\simeq 1$ with 0·05 *M* EDTA (disodium salt), with methylthymol blue or xylenol orange as indicator. Organic compounds containing bismuth are oxidised by boiling with a mixture (1:1) of $HClO_4$ (70%)

and HNO_3 (35%). Basic bismuth salicylate, basic bismuth gallate, basic bismuth tribromophenoxide, and basic bismuth iodogallate were analysed by this procedure. Owing to its high accuracy the method has been recommended for the new edition of the Czechoslovak Pharmacopoeia. J. VOLKE

See also Abstracts—**3587**, New reagent for organic bases. **3771**, Potentiometric titration of sulphonamides. **3782**, Determination of tartrates. **3790**, Determination of isoniazid. **3796**, Determination of piperidinomethyl*cyclo*hexanone. **3807**, Determination of thymol in thyme oil. **3826**, Detection and determination of chlorpromazine. **3827**, Determination of tolbutamide. **3862**, Paper-chromatographic separation of oestrogens. **3915**, **3916**, Determination of vitamin K_1. **3923**, Determination of tocopherol.

Food

3893. **Determination of nitrates in the milling industry.** S. Klein (Inst. Technol. of Milk, Fats and Meal, High-School of Chem. Technol., Prague). *Průmysl Potravin*, 1958, **9** (2), 110–111.—The method consists in the nitration of 2:4-xylenol and distillation of the resulting nitroxylenol into an alkaline medium; the yellow colour of the product is measured at 430 mμ. Organic compounds and chlorides do not interfere and the method is suitable for the control of meal and meal products. *Procedure for meal*—Mix the sample containing $\geq$ 3 mg of NO_3^- with H_2O (20 ml), dilute to 50 ml and measure 20 ml into a 50-ml flask. Nitrites can be removed by bubbling the soln. with CO_2 for 10 min. after the addition of methanol (1 ml) and 5 *N* H_2SO_4 (1 ml). Add 2 *N* NaOH (7 to 8 ml, in the absence of nitrites only 2 to 3 ml) and precipitate proteins by means of trichloroacetic acid (20%) (10 ml). Dilute to vol., filter through a paper filter and measure 5 ml of the filtrate into a 250-ml flask; add H_2SO_4 (85%) (15 ml) satd. with Ag_2SO_4 (about 50 g of Ag_2SO_4 in 1 litre of H_2SO_4), cool to 35°, add xylenol (1 ml), close the flask and maintain it at 35° for 30 min. Dilute the contents with H_2O (100 ml) and distil into a 50-ml flask containing 10 ml of a 10% soln. of Na_2SO_3 in 2 *N* NaOH. Measure the extinction and compare with a calibration curve. The average error is $\pm$ 3%.
J. ZÝKA

3894. **The use of EDTA in the determination of complex sugars in molasses.** J. P. Shukla, S. C. Jolly and K. A. Prabhu (National Sugar Inst., Kanpur, India). *Naturwissenschaften*, 1958, **45** (4), 84–85.—EDTA does not interfere in the Somogyi method for the assay of reducing sugars. Higher reducing values are obtained in the presence of EDTA as the EDTA removes Ca, Fe, etc., with which the molasses sugars are complexed.
E. KAWERAU

3895. **Colorimetric estimation of malathion residues in animal products.** M. V. Norris, E. W. Easter, L. T. Fuller and E. J. Kuchar (Stamford Lab., American Cyanamid Co., Conn.). *J. Agric. Food Chem.*, 1958, **6** (2), 111–114.—The technique previously reported for the determination of malathion [S-1:2-di(ethoxycarbonyl)ethyl dimethyl phosphorothiolothionate] in plant material (by alkaline decomposition in ethanol - CCl_4 soln. to give Na dimethylphosphorodithioate, conversion of this into a copper complex, and extraction of the complex into CCl_4 to give a yellow colour) (*cf.* Norris *et al.*, *Anal. Abstr.*, 1954, **1**, 2852) is adapted for use with animal products. Separate methods are described for determinations of malathion in fat, meat, liver, milk, egg white, and egg yolk, in concentrations of 1·0, 0·5, 1·0, 0·02, 0·5 and 0·5 p.p.m., respectively. M. D. ANDERSON

3896. **Chemical determination of paralytic shellfish poison in clams.** E. F. McFarren, E. J. Schantz, J. E. Campbell and K. H. Lewis (Robert A. Taft Sanitary Engng Center, U.S. Pub. Health Service, Cincinnati, Ohio). *J. Ass. Off. Agric. Chem.*, 1958, **41** (1), 168–177.—The determination depends on the selective adsorption of the poison on an Amberlite XE-64 cation-exchange resin, its elution with aq. trichloroacetic acid, and the application to the eluate of the Jaffé test. The ground shellfish-meat is boiled with 0·5 *N* trichloroacetic acid and the filtered extract, adjusted to a pH of 5·0 to 5·2, is passed through the column, which is then washed with McIlvaine's buffer solution followed by water. The poison is next eluted at pH $< 4{\cdot}5$ with 0·5 *N* trichloroacetic acid, which is then removed with ether, and the acid-free eluate is treated with picric acid and NaOH. After 20 min. at 38° the absorbance at 510 mμ is read; that of another aliquot is read after 15 min. at 100°. Under the latter conditions the absorbance of interfering material is 5 times as great as under the former conditions, whereas the absorbance of the purified poison decreases to about one-third. An equation for calculating the amount of poison present in an eluate containing variable amounts of interfering material has been developed. In the region 100 to 150 μg per 100 g the results are higher and more accurate than those afforded by bioassay.
A. A. ELDRIDGE

3897. **Centrifugal tube for extracting butyl alcohol in the phosphatase test.** L. Murthy and E. O. Herreid (Illinois Univ., Urbana, U.S.A.). *J. Dairy Sci.*, 1958, **41** (2), 315.—The construction of a special glass centrifuge tube is described.
W. H. C. SHAW

3898. **Determination of the total nitrogen in stored milk by nesslerisation and by the macro-Kjeldahl methods.** L. Murthy and E. O. Herreid (Illinois Univ., Urbana, U.S.A.). *J. Dairy Sci.*, 1958, **41** (2), 314–315.—From a study of the two methods it is concluded that direct nesslerisation after acid digestion of the sample gives satisfactory results on fresh milk, but not on sterilised milk subsequently stored at elevated temperatures; the discrepancies are discussed. W. H. C. SHAW

3899. **Application of electrophoresis on paper to the estimation of alpha-, beta- and gamma-casein.** H. F. Macrea and B. E. Baker (McGill Univ., Quebec, Canada). *J. Dairy Sci.*, 1958, **41** (2), 233–240.—The three components of casein are separated by paper electrophoresis and are estimated from densitometer readings on the paper after treatment with a suitable dye. Calibration curves are prepared with the dyes. Correction factors are necessary because the components do not bind the dyes in proportion to their concn. Results for the three components were 60%, 32% and 9%, respectively; these figures agree well with those given by protein-N determinations. W. H. C. SHAW

3900. **Polarographic determination of benzaldehyde in the juice of stewed fruit.** C. Párkányi (Control Inst. of Food Industry, Prague). *Průmysl*

Potravin, 1958, **9** (2), 111–112.—*Procedure*—Steam-distil 25 ml of a suitable amount of the sample into a 50-ml flask, dilute with phosphate buffer soln. (pH 6·8) and mix. Measure 2 ml into the polarographic vessel, add phosphate buffer soln. (pH 6·8) (0·3 ml), remove oxygen and register the polarographic wave from − 0·8 V. Compare with a standard soln. of benzaldehyde. J. ZÝKA

3901. **Ontogenesis of gramine in barley seedlings.** V. E. Tyler, jun. (Coll. of Pharm., Univ. of Nebraska, Lincoln, U.S.A.). *J. Amer. Pharm. Ass., Sci. Ed.*, 1958, **47** (2), 97–98.—A paper-chromatographic technique is used to separate gramine (**I**) from other basic compounds present in barley seedlings. It is shown that **I** is synthesised and stored in the plant shoot. *Procedure*—Prepare an ethanolic extract of the powdered plant material and transfer a concentrated aliquot (containing $\leqslant 5$ μg of **I**) to Whatman No. 1 paper. Develop by the ascending technique with 10% aq. $(NH_4)_2SO_4$ soln. for 5 hr. Dip the dried paper in Munier's reagent. Average R_F values are—**I**, 0·64; hordenine, 0·78; choline, 1·00.
A. R. ROGERS

3902. **Measurement of enzymic amylolytic activity. Group B. I. Chemical methods.** H. Wildner and G. Wildner (Ireks Forschungsinst. für Gärungswissenschaft, Kulmbach, Germany). *Brauwissenschaft*, 1958, **11** (2), 35–42.—Detailed descriptions are given of published methods for the determination of maltose formed by the action of β-amylase, the diastatic activity of malt, barley, and malt extracts, amylase in cereals and cereal products, α-amylase in rye meal and of limit-dextrinase in malt extract. Methods for the use of potato-starch as a substrate for the determination of diastatic activity, and for the activation of inactive amylase by means of cysteine are also described.
P. S. ARUP

3903. **Colorimetric determination of the obscuration in proof of straight whiskies.** R. A. Nelson, M. J. Pro and A. P. Mathers (Alcohol and Tobacco Tax Div. Lab., Internal Revenue Service, Washington, D.C., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1958, **41** (1), 139–140.—The obscuration in proof due to dissolved solids may be determined as a function of colour. Whiskies, treated with H_3PO_4 to form a colourless complex with any Fe present, were examined spectrophotometrically at 430 mμ. The solids (g per 100 litres) were plotted against absorbancies; 1 degree in proof is represented by 250 g of solids per 100 litres or 1·787 absorbance. Tabulated results show that obscuration values based on colour are as reliable as, and usually more precise than, those based on the determination of solids. A. A. ELDRIDGE

3904. **Colorimetric determination of vanillin.** D. T. Englis, G. Meerman and L. A. Wollermann (Dept. of Chem. and Chem. Engng, Univ. of Illinois, Urbana, U.S.A.). *J. Ass. Off. Agric. Chem.*, 1958, **41** (1), 203–206.—Vanillin soln. containing 5 ml of the Folin - Denis reagent and the equivalent of 1·25 g of Na_2CO_3 in a soln. of final volume 100 ml give a satisfactory colour value in 30 min. The colour change after 30 min. is slow. If the reactants are near the final required volume when mixed, it matters little whether the Na_2CO_3 is added first or last. A. A. ELDRIDGE

3905. **Losses from the use of the lead reagent in the analysis of vanilla extracts.** D. T. Englis, G. Meerman and L. A. Wollermann (Dept. of Chem. and Chem. Engng, Univ. of Illinois, Urbana, U.S.A.). *J. Ass. Off. Agric. Chem.*, 1958, **41** (1), 197–203.—Losses of vanillin resulting from the clarification of vanillin extracts with Pb acetate have been determined by ultra-violet spectrophotometry of the filtrate after removal of dissolved lead by means of Na_2HPO_4. The loss is reduced if the Pb acetate employed in the official (A.O.A.C.) method is added to the sample at a dilution of 480 ml instead of 80 ml before final dilution to 500 ml and filtering. The loss of vanillin in the analysis of an extract is greater than that encountered in treatment of the same amount of pure vanillin, losses of 6% and 3·2%, respectively, being reported. A. A. ELDRIDGE

3906. **Detection of Naphthol yellow and other yellow colourants in "Dijon" mustard.** P. P. Legrand. *Ann. Falsif.*, 1958, **51**, 5–18.—Methods involving paper chromatography, paper electrophoresis and column chromatography have been developed for the separation, purification and identification of Naphthol yellow in "Dijon" mustard, the experimental conditions of which vary with the amount of colouring matter present. The techniques of paper electrophoresis and chromatography were applied to highly or moderately coloured mustards. Weakly coloured mustards may be analysed by chromatography on an alumina column. S. M. MARSH

3907. **Detection of alkali refining of rancid lard.** J. Wurziger and E. Lindemann. *Fette, Seif., Anstrichmitt.*, 1958, **60** (2), 99–103.—When rancid lard is refined by alkali treatment (possibly accompanied by treatment with bleaching-earth and deodorisation) many of the characteristics indicative of rancidity, such as Lea and acid values, are reduced to acceptable values. The keto-acid content remains practically unchanged and this can be used to detect rancid lard that has been refined for illegal (in the German Federal Republic) sale as edible lard. The keto-acid content is determined by heating the lard with ethanolic KOH and measuring the extinction of the brown colour produced. *Procedure*—Boil a weighed sample (2 g) of filtered lard under reflux (or in a beaker covered with a watch-glass) for exactly 2 min. with 10 ml of colourless 0·5 *N* ethanolic KOH. Wash the reaction mixture into a calibrated flask with 96% ethanol, add 3 ml of water, cool rapidly to 20° and make up to 25 ml with 96% ethanol. Filter if necessary. After 10 min. (including 2 min. refluxing) measure the extinction of the soln. in an Elko photometer with a 1-cm cell and S47E filter; use an 8% soln. of the sample in ether as blank. Genuine lard shows $E^{1\%}_{1cm}$ values from 0·13 to 0·30; values $> 0{\cdot}40$ are suspect. Antioxidants must be absent. E. HAYES

3908. **Determination of arachis oil in edible oils by partition chromatography on filter-paper.** B. Briski and A. Brodarec (Central. Higijenski Zavod, Zagreb, Yugoslavia). *Kem. u. Ind., Zagreb*, 1958, **7** (1), 1–12.—The method described is based on the composition of the high-molecular fatty-acid fraction of edible oils, in particular the presence of behenic and lignoceric acids in amounts greater than that of the arachidic acid in the high-molecular fatty-acids fraction of refined arachis oil. The arachidic acid itself is not specific to arachis oil and is found in most edible oils. The free fatty acids are separated from the saponified oil and the saturated fatty acids are then separated from

the unsaturated ones by pptn. with Pb acetate. The high-molecular saturated fatty acids are then crystallised at a low temp. in 90% ethanol. A lipophilic filter-paper saturated with kerosine is prepared and a toluene soln. of the separated high-molecular fatty acids is chromatographed ascendingly with 96% acetic acid saturated with the same kerosine. The chromatograms are treated with Cu acetate and $K_4Fe(CN)_6$ and the various fatty acids become visible as greenish copper soaps, followed by brown spots of copper ferrocyanide. Several examples and reproductions of the chromatograms are given and the influence of the various factors is discussed. The melting-points of the crystallised mixtures of the isolated high-molecular fatty acids were also studied, but the paper-chromatographic method appears to be simpler for practical analytical application. It has been applied to the checking of mixtures of arachis oil with olive, sunflower and other refined edible oils, and is suitable for the analytical control of edible oils on an industrial scale. A. GROCHOWSKI

3909. **Oxidation of fats in emulsions. I. Determination of the peroxide value in emulsions.** P. Finholt and G. Hopp (Pharm. Inst., Oslo Univ., Norway). *Medd. Norsk Farm. Selsk.*, 1957, **19** (10), 163–173.—Factors that influence the Wheeler method for the determination of peroxide values (*cf. Oil & Soap*, 1932, **9**, 89) were investigated and a recommended procedure is described. *Procedure*—A 1-g sample of fat or oil, or a wt. of emulsion containing 1 g of fat, is mixed successively with 20 ml of $CHCl_3$ (warming if necessary), 30 ml of glacial acetic acid (cooling if necessary) and 1 ml of satd. KI soln.; the mixture is set aside for 3 min. in the dark at room temp.; 100 ml of water is added, and the mixture is titrated with 0·01 *N* $Na_2S_2O_3$, with starch as indicator. In the blank determination the amount of water present in the initial sample is added to the $CHCl_3$, acetic acid and KI soln. before dilution with 100 ml of water and titration. [*Cf. Riechstoffe u. Aromen*, 1958, **8** (1), 15–17.] H. L. WHITEHEAD

3910. **Constants of the total fatty acids from various animal and vegetable sources. Their utilisation for the recognition of esterified oils.** L. Matarese (Dogane e I. I., Bologna, Italy). *Olii Min.*, 1958, **35** (2), 39–46.—The differentiation, by analysis, of genuine olive oils from oils prepared by esterification of fatty acids from other oils or of acid oils is considered. To assist in the recognition of the source of the acids from the esterified oils, the constants (acid val., iodine val., refractive index, mol. wt. and solidification point) of the total acids from a range of edible oils and animal oleins have been determined. L. A. O'NEILL

3911. **Quantitative determination of unsaturated fatty acids on filter-paper.** G. Schmidt (Inst. f. angew. Zoologie, Univ., Würzburg). *Naturwissenschaften*, 1958, **45** (2), 41.—The methyl esters of unsaturated fatty acids form addition products with mercury salts. The methyl esters are produced either by transesterification of the glyceride esters of the total fatty acids by methanol, or by treatment of the free fatty acids with diazomethane. To every one part of the fatty acid methyl ester, five parts of mercuric acetate are added in 10 vol. of abs. methanol and boiled under reflux for 2 hr. The methanol is then removed completely by distillation and the residue is taken up in 50 vol. of light petroleum. Excess of mercuric acetate is removed by filtration, and the light petroleum soln. is spotted on the paper, which has been impregnated with tetrahydronaphthalene. The chromatogram is developed with 70% methanol - acetic acid (3:2, by vol.) by ascending technique for 10 to 12 hr. For quant. evaluation, the bound mercury salt is converted into the sulphide by first hanging the paper in an atmosphere of HCl vapour for 6 hr., then in an atmosphere of H_2S for 12 hr. The spots are measured by passing the paper through a densitometer and plotting the extinction curve. The individual components of the curve are obtained by planimetry and the absolute values are deduced from calibration curves. E. KAWERAU

3912. **The losses of vitamin A during chromatography.** G. Lambertsen and O. R. Braekkan (Gov. Vitamin Lab., Norwegian Fisheries Res. Inst., Bergen, Norway). *Acta Chem. Scand.*, 1958, **12** (2), 360–361 (in English).—The 10% loss of vitamin A during chromatography on alumina is reduced to about 2% by the incorporation of γ- or δ-tocopherols in the proportion of 200 μg of tocopherols per 1000 i.u. of vitamin A. A soln. of the unsaponifiable portion of arachis oil or soya-bean oil should be added to the vitamin-A fraction before chromatography of medium- and high-potency oils. A. R. ROGERS

3913. **Revised thiochrome procedure for determination of thiamine in cereal products.** W. G. Bechtel and C. M. Hollenbeck (Amer. Inst. of Baking, Chicago, Illinois). *Cereal Chem.*, 1958, **35** (1), 1–14.—The procedures described in "Cereal Laboratory Methods" (6th Ed., Amer. Ass. Cereal Chemists, St. Paul, Minnesota, 1957) are modified by (*i*) extracting the sample without drying it, (*ii*) extracting with 0·1 *N* H_2SO_4 instead of 2% acetic acid, (*iii*) adjusting the enzyme level and temp. to give the most convenient digestion time, and (*iv*) purifying (when necessary) on a column of Decalso only 3 cm high, so that the Decalso can be discarded to avoid the need for purifying and reactivating. A rapid method is given for activating Decalso. In 3 series of collaborative tests on a wide variety of cereal products, the modified procedure gave results at least equal in precision to those of other methods. M. D. ANDERSON

3914. **Liberation and determination of riboflavine in natural feeding-stuffs.** V. M. R. Murthy, R. N. Burroughs, B. L. Reid and J. R. Couch (Dept. of Poultry Sci. and Biochem. and Nutrition, Texas A & M College System, College Station, U.S.A.). *J. Agric. Food Chem.*, 1958, **6** (2), 129–130.—Simplified methods found to be satisfactory for the extraction of riboflavine from feeding-stuffs for routine determinations were (*i*) suspension of the sample in water, steaming in an autoclave for 15 min., cooling suddenly, centrifuging, and determining the riboflavine in the supernatant liquid by assay with *Lactobacillus casei*; and (*ii*) a similar method, in which 0·06% HCl is used instead of water, with determination of the riboflavine by fluorimetry. Recovery of added riboflavine was 97 to 100%. Agreement with the standard AOAC method was good. M. D. ANDERSON

3915. **A sensitive and stable colour reaction for the quantitative determination of vitamin K_1.** K. Schilling and H. Dam (Polytech. Inst., Copenhagen, Denmark). *Acta Chem. Scand.*, 1958, **12** (2), 347–348 (in English).—Dissolve the sample

(containing $\simeq$ 250 μg of vitamin K_1) in ethanol (3 ml), and mix with 1 ml of an 80%-saturated ethanolic soln. of 5-imino-1:2:4-dithiazolidine-3-thione. Add N aq. KOH (1 ml), place in a water bath at 50° for 10 min., cool rapidly and measure the extinction at 410 mμ against a reagent blank. The colour is stable for several hours. Tocopherol quinone gives only a slight colour when treated similarly; 2-methyl-1:4-naphthaquinone gives a red colour with max. absorption at about 440 mμ.

A. R. ROGERS

3916. **Colorimetric estimation of vitamin K_1 in plant material.** K. Schilling and H. Dam (Polytech. Inst., Copenhagen, Denmark). *Acta Chem. Scand.*, 1958, **12** (2), 348–349 (in English).—Extract the sample containing vitamin K_1 (**I**) with light petroleum and pass the extract through a column of activated calcium hydrogen orthophosphate. Elute with light petroleum until the β- and α-carotene bands have passed through; chlorophylls and xanthophylls remain on the column. Evaporate the eluate to dryness at 20° under reduced pressure and dissolve the residue in benzene (3 ml). Add 1·5 ml of an 80%-saturated ethanolic soln. of 5-imino-1:2:4-dithiazolidine-3-thione and heat in a water bath at 50° for 5 min. with a 4 N soln. of KOH in methanol (0·5 ml). Cool rapidly and extract with a mixture of light petroleum (10 ml) and 50% ethanol (2 ml) and then with light petroleum (10 ml). Measure the extinction of the aq. phase at 410 mμ against a reagent blank. No loss of **I** was apparent in recovery experiments, and the content of **I** in a sample of lucerne meal estimated by this method was in good agreement with biological determinations.

A. R. ROGERS

See also Abstracts—**3776**, Determination of methanol in ethanol and fruit brandies. **3778**, Determination of glucose. **3806**, Determination of Fe in gelatin. **3836**, **3837**, Determination of vitamin A. **3923**, Determination of tocopherol. **3944**, Apparatus for detection of sugar in boiler-feed water. **3953**, Automatic recording saccharimeter.

Sanitation

3917. **Determination of small amounts of E605 (parathion, diethyl *p*-nitrophenyl phosphorothionate) and methyl E605 (dimethyl *p*-nitrophenyl phosphorothionate) [parathion-methyl] in air and dust.** W. Pilz (Farbenfabriken Bayer A.-G., Leverkusen, Germany). *Mikrochim. Acta*, 1958, (3), 383–394 (in German).—The absorption of parathion (**I**) and parathion-methyl (**II**) from air was studied in a special apparatus (shown diagrammatically), and the absorption in 2-methoxyethanol - N NaOH (1:1), at a flow rate of 1 litre per min., was found to be quant. Alkaline hydrolysis of **I** and **II** yields *p*-nitrophenol, which may be reduced to *p*-aminophenol. By coupling this with another phenol, indophenol blue is obtained, which may be determined photometrically, thus providing a possible method for determination of **I** and **II**. Results of a systematic study of the reactions are described, and it is shown that unsubstituted phenol is the most satisfactory phenol for coupling, and also that the indophenol reaction is specific. Details of analytical procedures for the determination of **I** and **II** in air, in dusts and in coloured materials are given. The maximum error for the determination in air is $\pm$ 3·50% and the lowest detection limit is 0·0015 p.p.m.

S. M. MARSH

3918. **Conditions for the conservation and subsequent determination of sulphides in natural waters.** B. A. Skopintsev and A. V. Karpov. *Gidrokhim. Materialy*, 1957, **26**, 230–236; *Ref. Zhur., Khim.*, 1958, Abstr. No. 7626.—Take an aliquot of the water to be analysed (in the absence of dissolved O) into a calibrated flask, through which CO_2 has first been passed. Introduce on to the surface of the water in the neck of the flask 7 drops of a soln. of Zn acetate (40 g in 100 ml), and mix. Flush out a conical flask, fitted with a ground glass stopper, with CO_2, and add 2 ml of H_2SO_4 (1:3), 10 ml of 0·02 N iodine or KIO_3 and 0·5 g of KI, and mix. Add to this flask the contents of the calibrated flask, including the ZnS, and titrate the excess of iodine with 0·01 N $Na_2S_2O_3$ in the presence of starch. On keeping the conserved sulphides for 9 to 96 hr. the results obtained are 95 to 98% of the results by the standard method. A method is also described for determining H_2S in water containing dissolved O.

C. D. KOPKIN

3919. **Conductimetric titration of sulphates in natural waters.** M. P. Belsova and P. A. Kryukov. *Gidrokhim. Materialy*, 1957, **26**, 190–206; *Ref. Zhur., Khim.*, 1958, Abstr. No. 7623.—With $>$ 10 milli-equiv. of SO_4^{2-} per litre, pass a known vol. of the water to be analysed, containing 0·1 to 2 milli-equiv. of SO_4^{2-} per litre, through a column of 3 g of cationite SBS or SBSR (H form) previously washed with double-distilled water, at a rate of 1 ml per min., and wash the column with water. Collect the eluate and wash liquors and dilute to 50 ml. Place an aliquot in a conductimetric vessel, pass CO_2-free air for 10 min. and neutralise with 0·1 N NaOH. With $<$ 10 milli-equiv. of SO_4^{2-} per litre, add to the neutralised soln. an equal vol. of ethanol. Place the vessel in a thermostat and titrate conductimetrically on a precision instrument with $Ba(NO_3)_2$ soln., the concn. of which is 10 times as great as that of SO_4^{2-}. With $<$ 10 milli-equiv. of SO_4^{2-} per litre, destroy HCO_3^- before the titration. The time for a determination is 45 min., and the mean deviation of results from those of the gravimetric method is 1·1% with $>$ 1 milli-equiv. of SO_4^{2-} per litre, and 5·4% with $<$ 1 milli-equiv. of SO_4^{2-} per litre. The presence of a 5-fold quantity of NaCl (compared with the quantity of SO_4^{2-}) does not cause interference; the presence of twice this quantity of $CaCl_2$ leads to an error of $\simeq$ 10%.

C. D. KOPKIN

3920. **The direct determination of ammonia in sea water, with notes on nitrate, copper, zinc and sugars.** W. R. G. Atkins. *J. Cons. Int. Explor. Mer*, 1957, **22**, 271–277.—A colorimetric method for the determination of ammonia in sea water, with chloramine T and a pyridine - pyrazolone reagent, is described and details of the procedure are given. Methods for the determination of NO_3^-, Cu, Zn and sugars in sea water are compared. The most satisfactory reagent for the determination of NO_3^- is diphenylbenzidine but, as this involves the use of conc. H_2SO_4, reduction of the NO_3^- to NO_2^- with a quartz - mercury arc, followed by the Griess - Ilosvay reaction at pH $\not<$ 9, is suggested. A comparison is made between the various organic solvents used to determine Cu, and Na diethyldithiocarbamate and extraction with CCl_4 is recommended. The most sensitive reagent for the determination of Zn was found to be *p*-dimethylaminostyryl-β-naphthiazole methiodide, although Na diethyldithiocarbamate is fairly satisfactory.

Both are preferable to ferrocyanide. Sugars, such as arabinose, are most satisfactorily determined with N-ethylcarbazole. WATER POLLUT. ABSTR.

3921. **Gallium in sea water.** F. Culkin and J. P. Riley (Dept. of Oceanography, Univ., Liverpool). *Nature*, 1958, **181**, 180.—Gallium was pptd. from 5-litre samples in 93 ± 5% yield with ferric hydroxide as carrier. The ferric hydroxide was dissolved in HCl, reduced with $TiCl_3$ and the Ga was extracted with di*iso*propyl ether. After evaporation of the ether, Ga was determined spectrophotometrically by a modification of the rhodamine B method of Onishi and Sandell (*Anal. Abstr.*, 1956, **3**, 58). The average concn. found was ≃ 0·03 μg per kg. N. E.

3922. **Germanium in sea water.** J. D. Burton and J. P. Riley (Dept. of Oceanography, Univ., Liverpool). *Nature*, 1958, **181**, 179–180.—Addition to 5-litre samples of ferric alum and pptn. of ferric hydroxide carried down > 97% of the Ge present. The ppt. was dissolved in conc. HCl and the Ge extracted from the soln. with CCl_4, back-extracted with H_2O and determined spectrophotometrically with phenylfluorone. The average concn. found was ≃ 0·06 μg per kg. N. E.

Agriculture and Plant Biochemistry

3923. **Chromatographic separation and estimation of certain pasture lipids. II. Tocopherol.** N. A. Worker (Biochem. Dept., Massey Agric. Coll., Univ. of N. Zealand, Palmerston North). *J. Sci. Food Agric.*, 1958, **9** (2), 122–124.—A method for the routine determination of tocopherol in pasture samples is described. *Procedure*—Extract the sample (5 oz fresh or 1 g dried) as described in Part I (*cf. Anal. Abstr.*, 1957, **4**, 4163). Evaporate a 30-ml aliquot of the filtrate to dryness at 60°, re-dissolve the residue in 1% v/v ethanol in light petroleum (boiling range 40° to 60°) (solvent *A*) (2 to 3 ml) and transfer to a prepared column of MgO (heavy grade). Elute with solvent *A* (40 ml), evaporate the eluate to dryness, re-dissolve the residue in light petroleum (2 to 3 ml) and transfer to a prepared column of Al_2O_3 (Brockman Grade III). Elute with 1% v/v acetone in light petroleum (10 ml). Recover the adsorbed tocopherol by eluting with 8% v/v ethanol in light petroleum (25 ml). Evaporate the eluate to dryness and re-dissolve the residue in ethanol (5 ml). To 4 ml of the ethanolic soln. add 2:2′-dipyridyl reagent (0·25% w/v in ethanol) (0·5 ml) and $FeCl_3$ (0·1% w/v in ethanol) (0·5 ml) and determine the absorption at 520 mμ. Obtain the tocopherol content by reference to a standard graph. Recoveries of 95 to 103% are quoted. H. B. HEATH

3924. **New qualitative reaction of 5-hydroxy-1:4-naphthaquinone [juglone], and its use in colorimetric analysis.** L. N. Alsenberg. *Trudy Kishinevsk. S.-Kh. Inst.*, 1956, **11**, 29–36; *Ref. Zhur., Khim.*, 1958, Abstr. No. 7703.—To an ethanolic soln. of juglone add 1 ml each of a 3% aq. soln. of $Ni(NO_3)_2.6H_2O$ and a 0·1% $NaHCO_3$ soln. The nickel carbonate reacts with the juglone and forms an intensely violet-coloured, stable, internally-complexed nickel salt of formula $C_{20}H_{10}O_6Ni$. The sensitivity with a 0·15% ethanolic soln. of juglone is 1 in $2·5 \times 10^5$ (in the absence of Cu, Fe^{2+}, Co or Cr^{2+}). This reaction may be used for the photometric determination of juglone, comparison being made with standards prepared from an ethanolic soln. of freshly sublimed juglone (m.p. 154°). C. D. KOPKIN

3925. **Precipitation of resin acids by salts of hexamethylenediamine.** L. Desalbres and R. Dubéarnès (Lab. de la Soc. de Dériv. Résiniques et Terpéniques). *Bull. Soc. Chim. France*, 1958, (2), 229–230.—Pure abietic acid and dehydro- and dihydro-abietic acids and the crude acids from maritime pine resin, from maritime pine rosin and from tall oil are dissolved in *iso*butyl methyl ketone and pptd. quantitatively at 70° with hexamethylenediamine in the same solvent. The acids are recovered from the salts by suspending them in *iso*butyl methyl ketone and treating them with aq. 10% HCl. E. J. H. BIRCH

3926. **Gibberellic acid and its potassium salt. A short study by paper chromatography.** L. C. Mitchell (Div. of Food, Food and Drug Admin., Dept. of Health, Education and Welfare, Washington, D.C., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1958, **41** (1), 182–185.—Gibberellic acid and its potassium salt have been shown by paper chromatography to contain two impurities. The mobile solvent used comprises *n*-butanol (25 ml), acetone (25 ml), *tert.*-amyl alcohol (25 ml), aq. NH_3 (10 ml) and water (to 100 ml). The gibberellic anion (R_F value 0·51 to 0·58) and the impurities (R_F values 0·13 to 0·21 and 0·65 to 0·71) were detected by means of a chromogenic agent prepared by mixing 4 parts of 1% (w/v) aq. KIO_4 with 1 part each of 1% (w/v) aq. $KMnO_4$ soln. and 1% (v/v) H_2SO_4. After 5 to 7 min., excess of permanganate is washed out with water and the chromatogram is air-dried. Over-spraying with a second chromogenic agent (benzidine 0·4 g, aq. NH_3 10 ml, and water 30 ml, diluted to 200 ml with ethanol) is advantageous. A. A. ELDRIDGE

3927. **A modified procedure for determining maleic hydrazide residues in plant material.** C. Anglin and J. H. Mahon (Food and Drug Lab., Dept. of Nat. Health and Welfare, Ottawa, Ontario, Canada). *J. Ass. Off. Agric. Chem.*, 1958, **41** (1), 177–182.—Wood's method (*cf. Anal. Abstr.*, 1954, **1**, 1125) has been modified to avoid certain difficulties, which include decomposition of some of the maleic hydrazide by NaOH used to eliminate interfering materials, and frothing during distillation. The pulverised plant material is heated at 190° with NaOH, zinc and water and the mixture is steam-distilled into the *p*-dimethylaminobenzaldehyde reagent. The red interfering material exhibits extinctions at 500 mμ and 455 mμ which are directly proportional; the *p*-dimethylaminobenzaldazine absorbs at 455 mμ but not at 510 mμ. The correction factor that can thus be applied must be determined for each type of material analysed. For tobacco, nicotine and other alkaloids must first be extracted with $CHCl_3$ at pH 4·6, and the usual correction factor must also be applied. Of 56 determinations, 45 afforded results for added maleic hydrazide within 1 p.p.m. Potatoes, presumably treated at the recommended level of application, contained 18 p.p.m. The adduct is distributed uniformly within the potato and is not destroyed by frying chips in oil. A. A. ELDRIDGE

3928. **Measurement of microgram amounts of chlorine in plant materials.** C. M. Johnson, R. P. Huston and P. G. Ozanne (Dept. of Soils and Plant

Nutrition, Univ. of Calif., Berkeley). *J. Agric. Food Chem.*, 1958, **6** (2), 114–118.—Work on Cl as a plant micro-nutrient required determinations on Cl-deficient plants containing as little as 35 μg of Cl per g. Methods developed were (*i*) an adaptation of the Conway micro-diffusion technique, enabling as little as 0·4 μg to be determined in soln. of ashed material, and (*ii*) the application to both aq. extracts and soln. of ash of Kolthoff and Kuroda's potentiometric titration of Cl to the apparent equiv. potential (*cf. Brit. Abstr. C*, 1952, 49). Pretreatment of samples with $Ca(OH)_2$ suspension or K_2CO_3 soln. before ashing is required to minimise losses of Cl during ashing. When the content of Cl is $\simeq$ 200 p.p.m., method (*ii*) applied to aq. extracts is rapid and convenient; for samples containing little Cl, ashing is preferable. Agreement between the two methods was good. Bromine and iodine are not distinguished from Cl.

M. D. ANDERSON

3929. **Thermogravimetry of soils.** I. Hoffman, M. Schnitzer and J. R. Wright (Chem. Div., Dept. Agric., Ottawa, Canada). *Chem. & Ind.*, 1958, (9), 261.—The value of thermogravimetric measurements in assessing the constitution of a soil is discussed. H. B. HEATH

3930. **Determination of total sulphur in soils, minerals, plants and organic compounds.** R. Kh. Aidinyan. *Pochvovedenie*, 1957, (9), 49–59; *Ref. Zhur., Khim.*, 1958, Abstr. No. 21,191.—The quant. extraction of S from soils, minerals, plants and organic compounds is based on heating the sample with $KClO_3$, which decomposes with evolution of O, causing oxidation of S to the soluble SO_4^{2-}. Mix 1 to 1·5 g of finely ground sample with 2 g of $KClO_3$ and 8 g of pure sand (to moderate the course of the reaction) in a quartz tube (length 18 cm, diam. 2·5 cm), which is joined to a glass tube as receiver by glass tubing. More than half fill the receiver with water and add one or two drops of 30% H_2O_2. The rubber bung in the receiver has a hole to allow escape of gas. Ignite the mixture over a gas burner, beginning at the end of the tube, and continue till no more sparks or bubbles are formed ($\simeq$ 10 min.). After the ignition, add to the cooled quartz tube two or three drops of HCl and add the contents of the receiver. Stir, filter on a heavy paper, wash the filter with 0·1 *N* KCl to give a total volume of filtrate of 200 or 250 ml, acidify the filtrate and precipitate SO_4^{2-} with 10% $BaCl_2$ soln. If for any reason there still remain black pieces of unignited material, then transfer the contents of the tube to a porcelain dish, add 0·2 g of $KClO_3$ and a few drops of HCl and evaporate on the water bath to dryness, repeating this operation several times, or treat once or twice with 2 or 3 ml of 30% H_2O_2. The method is universally applicable, rapid and simple, does not require complicated apparatus, and gives reliable results. C. D. KOPKIN

3931. **Colorimetric determination of molybdenum in soils and plants.** Yu. I. Dobritskaya. *Pochvovedenie*, 1957, (3), 91–100; *Ref. Zhur., Khim.*, 1958, Abstr. No. 21,176.—The thiocyanate method is modified. The influence of the method of decomposition of the soil, the concn. of HCl, Fe^{3+}, KSCN and NO_3^-, and the nature of the solvent is studied. It is established that instead of fusing the soil with a mixture of Na_2CO_3 and K_2CO_3 in a platinum vessel, it is more expedient to decompose it with HF. The sensitivity of the method is 0·5 μg of Mo in 12·5 ml of extract. C. D. KOPKIN

3932. **Use of phenylanthranilic acid in the determination of humus by the method of I. V. Tyurina.** V. N. Simakov. *Pochvovedenie*, 1957, **8**, 72–73; *Ref. Zhur., Khim.*, 1958, Abstr. No. 21,246.—Oxidise the sample of soil by boiling with chromic mixture according to Tyurina's method (*Pochvovedenie*, 1931, No. 5–6). Cool, add 3 or 5 drops of a 0·2% soln. of phenylanthranilic acid in 0·2% NaOH soln. and titrate with 0·2 *N* $(NH_4)_2SO_4.FeSO_4$ till the colour changes from cherry red to green. At the same time carry out a blank, with ignited soil. C. D. KOPKIN

3933. **Micro-determination of the medicaments furazolidone and nitrofurazone.** H. F. Beckman (State Chem. Lab., Texas Agric. Exp. Sta., College Sta., Texas). *J. Agric. Food Chem.*, 1958, **6** (2), 130–132.—Furazolidone [3-(5-nitro-2-furfurylideneamino-2-oxazolidone] (**I**) and nitrofurazone (5-nitro-2-furfuraldehyde semicarbazone) (**II**) are determined in feeding-stuffs by extraction with dimethylformamide and chromatography on alumina; **I** is eluted with dimethylformamide, and **II** with ethanol; on addition of KOH in 50% ethanol, a blue colour develops with **I**, and a pink-orange colour with **II**. Recovery was 95 to 102% for **I**, and 90 to 108% for **II**. 4-Hydroxy-3-nitrophenylarsonic acid, if present, is retained on the column. M. D. ANDERSON

3934. **Determination of total chlorine in pesticides by reduction with a liquid anhydrous ammonia - sodium mixture.** H. F. Beckman, E. R. Ibert, B. B. Adams and D. O. Skovlin (Texas Agric. Exp. Sta., Coll. Sta., U.S.A.). *J. Agric. Food Chem.*, 1958, **6** (2), 104–105.—Chlorine is removed from chlorinated pesticides by reduction with sodium in anhydrous NH_3, ether being added, if necessary, to ensure dissolution. After evaporation of solvents, and addition of water and HNO_3, Cl is determined by titration with $AgNO_3$ soln. Sulphur, if present, is removed by boiling with H_2O_2 after removal of solvents. The complete analysis takes 30 min. Values obtained for 6 pesticides were slightly higher by this method than by the sodium - *iso*propyl alcohol method. M. D. ANDERSON

3935. **Pyridine - alkali reactions in analysis of pesticides containing active halogen atoms.** H. P. Burchfield and P. H. Schuldt (Boyce Thompson Inst. for Plant Res., Yonkers, N.Y.). *J. Agric. Food Chem.*, 1958, **6** (2), 106–111.—Several independent analytical applications of the reactions of active halogen compounds with pyridine and alkali to give coloured products have been developed. Reaction mechanisms are discussed, and the underlying unity of the procedures is stressed. Methods based on this reaction are available for the quant. determination of such pesticides as the dichloro-*sym.*-triazines, 1-fluoro-2:4-dinitrobenzene, 3:4-dichlorotetrahydrothiophen-1:1-dioxide, and captan (N-trichloromethylthio*cyclo*hex-4-ene-1:2-dicarboxyimide); qualitative colour tests for other pesticides are described. M. D. ANDERSON

3936. **Detection and quantitative determination of parathion [and *p*-nitrophenol] in biological materials.** K. Erne (State Vet. Med. Inst., Stockholm, Sweden). *Acta Pharm. Tox., Kbh.*, 1958, **14** (2), 173–187.—The homogenised sample (10 g), mixed with 5 *M* H_2SO_4 (1 ml) and Hyflo Super-cel (20 g), is extracted with benzene in a Soxhlet apparatus. The benzene extract is chromatographed on acid alumina, interfering substances are

eluted with benzene, parathion (**I**) with diethyl ether, and *p*-nitrophenol (**II**), the main degradation product, with methanol. The ether extract is concentrated and purified by chromatography on paper impregnated with dimethyl sulphoxide. The appropriate area of the chromatogram, after development with *iso*octane, is cut out and eluted with ethanol. The ethanol is diluted and acidified, and **I** is reduced with zinc. After diazotisation and coupling with N-1-naphthylethylenediamine, **I** is determined from the extinction at 555 mμ. For the determination of **II**, the methanol extract is diluted and made alkaline, and lipids are extracted with diethyl ether. After acidification, **II** is extracted with diethyl ether and the concentrated extract is chromatographed on paper, which is developed with *n*-butanol saturated with 5 *M* aq. NH_3. The yellow area of the chromatogram is cut out and eluted with methanol. After addition of NaOH, **II** is determined from the extinction at 390 mμ. Detection of 0·2 μg of **I** or **II** per g of sample is possible and 1 μg per g can be determined. The recovery is $> 85\%$ and reproducibility within $\pm$ 7% of the mean.

W. H. C. SHAW

3937. **Identification of nitrothiophosphoric esters in toxicological tests.** M. Garnier and A. Wakli (Fac. Med. Pharm., Beirut). *Lebanese Pharm. J.*, 1957, **5**, 39–42 (in French).—*Procedure*—Extract tissue or viscera with acidified diethyl ether, evaporate the extract to dryness, add 10 ml of ethanol, 20 ml of H_2O, 2 ml of 6 *N* HCl, 1 g of liquid paraffin and 0·5 g of zinc, and maintain for 10 min. on a steam bath under reflux. Then cool the mixture slightly, filter on cotton, wash with 3 ml of H_2O, diazotise with 1 ml of 0·25% $NaNO_2$ soln., allow to stand for 10 min., add 1 ml of 2·5% $(NH_4)_2SO_4$ soln., set the mixture aside for 10 min., then add 1 ml of N-1-naphthylethylenediamine dihydrochloride. After 10 min. add 2 ml of 6 *N* HCl and 50 ml of ethanol and make up to 100 ml. The pink - peach to rose - violet colour attains a max. after 10 min., is very stable, and can be compared with prepared standards. 2-Naphthol was not selective as a coupling agent in this test.

CHEM. ABSTR.

See also Abstracts—**3681**, Determination of phosphate in calcium superphosphate. **3840**, Determination of raffinose and melibiose. **3895**, Determination of malathion. **3917**, Determination of parathion and parathion-methyl. **3945**, Micro-extractor for biological materials.

5.—GENERAL TECHNIQUE AND LABORATORY APPARATUS

General

3938. **New arrangement for agitation.** A. Salaün (Soc. d'Étude Chim. pour l'Ind. et l'Agric.). *Bull. Soc. Chim. France*, 1958, (2), 238–239.—An alternating pump is connected by a short wide tube to a "regulator-distributor" consisting of a reservoir 10 times the vol. of the pump cylinder and provided with outlets carrying taps. Each outlet can be connected to a tube dipping below the surface of the liquid to be agitated, so that at each stroke the liquid is sucked up into the tube and allowed to fall back at 60 to 90 strokes per min. Sizes of pump and tube for various amounts of liquid and shapes of vessel are listed. E. J. H. BIRCH

3939. **Automatic pipetting attachment for the Van Slyke manometric apparatus for carbon dioxide determinations.** D. Seligson and J. Marino (Grad. Sch. of Med., Univ. of Pennsylvania, Philadelphia, U.S.A.). *Clin. Chem.*, 1958, **4** (2), 120–126.—The pipette is made by straightening the waste tube of the extraction chamber, drawing a tip and then bending to an angle of 45°. Calibration is made with mercury. H. F. W. KIRKPATRICK

3940. **A fully-automatic, self-regulated water-distillation apparatus.** U. M. Weissenberg (Hebrew Univ., Jerusalem, Israel). *Lab. Practice*, 1958, **7** (2), 95–97.—In a modification of the electrically heated, all-Pyrex-glass, const.-level tap-water still designed by Post (Swiss Patent 267,693; *Chem. Abstr.*, 1951, **45**, 3538), the gradual electrolyte enrichment is eliminated so that there is no rise in conductivity and rate of distillation of the still-pot soln. The new apparatus is described and illustrated, and a simple formula expressing its performance under equilibrium conditions is given. An automatic device for ensuring a const. ratio of distillation to drainage rate with uniform continuous distillation at any required rate is also described. W. J. BAKER

3941. **A simple sublimation apparatus.** H. J. B. *M. & B. Lab. Bull.*, 1958, **3** (2), 26–27.—The construction and operation of this apparatus (illustrated) are described. It consists of a Pyrex-glass tube connected by pressure tubing to a vacuum pump, and heated by means of a copper or brass block encircling the tube. The operation can be scaled down to a semi-micro scale (10 to 50 mg). The apparatus can be used for short-path evaporative distillation. O. M. WHITTON

3942. **A continuous extraction apparatus.** J. C. O'Keeffe (Crawford Munic. Tech. Inst., Cork, Eire). *Lab. Practice*, 1958, **7** (2), 97–98.—An apparatus for the continuous extraction of a complex natural substance with boiling ethanol is described and illustrated. It has a greater capacity than the usual Soxhlet apparatus, whilst extraction takes place at the actual b.p. of the solvent and in the body of the apparatus itself. W. J. BAKER

3943. **Apparatus for the removal of salts from solutions of amino acids.** D. I. Semenov (Lab. of Biophysics, Urals Filial of the Acad. of Sci., USSR, Sverdlovsk). *Biokhimiya*, 1958, **23** (2), 296–299.—A modification of the apparatus of Consden *et al.* (*Biochem. J.*, 1947, **41**, 590) is described, which allows the removal of salts from soln. of amino acids in 5 to 7 min. Smaller vol. of soln. may be used (1 to 3 ml), back diffusion of anions is avoided, the temperature of the soln. does not rise above 25° or 30°, and the circulating water cannot pass into the soln. C. D. KOPKIN

3944. **The "Spaldinlab" Sugar-tektor.** W. H. Parker and G. M. Bond. *Int. Sugar J.*, 1958, **60**, 71–75.—A detailed description is given, with diagrams, of an apparatus for the continuous automatic detection of sugar in boiler-feed water, condensates, etc. Every 15 min., 100-ml water samples are taken from each boiler, treated with HCl, and boiled in a Vitreosil funnel to invert any sugar present. After adjustment of the pH with NaOH to $\simeq$ 11·0, triphenyltetrazolium chloride is added. In the presence of invert sugar a mauve ppt. of formazan is formed, and this passes into the inspection cell through which a beam of white light is directed, and this falls, via Calorex filters,

on to a photocell. The amount of ppt. is proportional to the amount of invert sugar. The sensitivity of the apparatus is ≃ 2 p.p.m. of sugar and provision is made for a meter calibrated in sugar concn. By the use of three Calorex filters, a red warning lamp on the boiler and an alarm bell can be actuated at the 5 p.p.m. level or less. SUGAR IND. ABSTR.

3945. **A micro-extractor for biochemical and chromatographic purposes.** R. Antoszewski (Inst. f. Pflanzenphysiol., Univ. Lodz, Poland). *Naturwissenschaften*, 1958, **45** (2), 42.—A narrow tube with a capillary opening is mounted into the mouth of a small Erlenmeyer flask with a side arm. The capillary end points downwards and reaches not quite to the bottom of the flask. The material to be extracted (*e.g.*, pollen, seeds, filter-paper) is inserted into this tube from the upper wider end and a few drops of solvent are added. The upper end of this tube is connected to a suction pump. A small volume of solvent, not sufficient to reach to the lower end of the capillary tube, is poured into the Erlenmeyer flask. When suction is applied, solvent vapour is drawn up through the capillary tube; this gives continuous agitation to the small volume of extractant in the tube as well as to the material, and extraction proceeds in this fashion. The side arm of the Erlenmeyer serves (*a*) to replenish the solvent should this become necessary and (*b*) to effect extraction in the atmosphere of a gas other than air. E. KAWERAU

3946. **An improvement in the technique of frontal analysis of mixtures by the use of divided columns.** P. P. Pillay, D. S. Rao, C. P. N. Nair and E. T. Varkey (Univ. of Kerala, Trivandrum, India). *Chem. & Ind.*, 1958, (9), 258–259.—A modified chromatographic-column packing is described and illustrated. The column of adsorbent is split into lengths of ≃ 15 cm, each length being separated by a plug of Pyrex-glass wool. In use, such columns have a zone of 0·5 to 1 cm of clear eluent between each wool plug above and adsorbent column below. Columns of any height can be built up without decrease in flow-rate, and channelling is avoided. H. B. HEATH

3947. **Adsorption chromatography on silica-treated paper.** J. B. Roberts (Colonial Microbiol. Res. Inst., Trinidad, B.W.I.). *Nature*, 1958, **181**, 338.—Whatman No. 1 paper is passed slowly through a 25% aq. soln. of commercial sodium silicate, drained for 30 min. and, while still wet, passed slowly through 3 *N* HCl. After draining for 30 min. and washing in running tap-water for 2 hr., the paper is hung up to dry overnight. Heating the dry paper for 15 to 30 min. at 100° before use activates the silica and secures better resolution. The mixture to be resolved, preferably in an organic solvent, is applied in the usual way, and the spots must be dry before chromatography, which is by the ascending technique, with light petroleum, benzene or $CHCl_3$ as solvent, the front being allowed to rise 10 to 15 cm. Reactive substances can be detected by their own specific reactions; unreactive ones are revealed as brown spots on a white background by dipping the dry chromatogram in 1·5% iodine in CCl_4 and allowing the excess to evaporate in a stream of air, these spots being intensified by dipping in 1% starch soln. and removing background blue by brief exposure to SO_2 gas. The procedure has been applied to mixtures of many organic compounds, including terpenes, leaf pigments, phenolic esters and ethers, and steroids. R. E. ESSERY

3948. **A recording dielectrometric method for column chromatography.** R. A. Grant (Natal Univ., Durban, S. Africa). *J. Appl. Chem.*, 1958, **8** (2), 136–140.—The instrumentation described enables colourless compounds to be detected and determined (approx.) in the eluate from a chromatographic column. The dielectric const., which generally changes with the composition of the soln., is measured continuously at const. temp. and is recorded on a chart. The separation of neutral amino acids (≃ 3 to 5 mg of each) on columns of cellulose powder with mixtures of *n*-propanol and water (15 to 30%) as solvent is reported. The min. concn. of amino acid to ensure a definite peak is ≃ 2 mg; the peak area is approx. proportional to the concn. W. J. BAKER

3949. **Flame-ionisation detector for gas chromatography.** J. Harley, W. Nel and V. Pretorius (Engng Res. Sta., S. African Iron & Steel Industrial Corp., Ltd., Pretoria). *Nature*, 1958, **181**, 177–178.—The sensitivity of the detector, which depends on the ionisation in a hydrogen flame of the eluate from a gas-chromatographic column, is such that full-scale deflection can be obtained on a 0 to 1-mA meter or recorder with an injected sample of 1 μl. The signal-to-noise ratio is better than 1000 to 1, and no base-line drift is observable over a wide range of operating conditions. The electrical output appears to depend linearly on the sample size as far as was tested up to 10 μl, and the reproducibility is limited only by injection precision. K. A. PROCTOR

3950. **Ion-exchange membranes in preparative and analytical chemistry.** E. Blasius and G. Lange (Tech. Univ., Berlin). *Z. anal. Chem.*, 1958, **160** (3), 169–188.—Apparatus for electrodialysis consisting of a block of 6 or 8 cells in series separated by ion-exchange membranes is described. The end cells contain copper electrodes and $CuCl_2$ soln. The remaining cells are filled in pairs with test soln. on the anode side and electrolyte on the cathode side of each pair. Dialysis is carried out at a constant voltage of 14·5 V. By the choice of membranes, pH range and complexing agents and taking advantage of differences in polarity, stability and mobility, quantitative separations can be achieved, and materials purified or prepared by double decomposition with analytical reagent purity. It is possible to prepare complex compounds of which the free base is unstable. G. BURGER

Optical

3951. **A lithium internal-standard flame photometer.** J. E. Amoore, D. S. Parsons and W. C. Werkheiser (Med. Res. Council Unit for Res. in Cell Metabol., Univ. of Oxford, England). *Biochem. J.*, 1958, **69** (2), 236–238.—The design and operation of a simple coal gas - air flame photometer, with Li as internal standard, are described. This internal standard compensates for interference by many substances, and gives greater precision and accuracy in the determination of Na and K by flame photometry. J. N. ASHLEY

3952. **A monochromator for u.v. microspectrometry.** H. Glubrecht (Inst. f. Strahlenbiologie, Tech. Hochschule, Hannover, Germany). *Naturwissenschaften*, 1958, **45** (2), 33.—The monochromator is a modification of that described by Joung (*J. Franklin Inst.*, 1871, **62**, 348), with an

additional mirror. By using a high-pressure mercury lamp the 265-mμ line can be isolated with an admixture of only 1·0‰ of stray light without the use of filters. E. KAWERAU

3953. **Automatic recording saccharimeter.** J. W. Gates (Nat. Phys. Lab., England). *Chem. & Ind.*, 1958, (7), 190–193.—A beam of plane-polarised light passes through a 10-cm DEDF glass rod that forms the core of an electromagnet. The 50 c.p.s. magnetic field induces an optical rotation alternating 3° either side of the unmodulated direction. The modulated beam passes through the sample cell and a second (5 cm) glass rod which forms the core of a d.c. electromagnet. After passing through an analyser, the beam is observed by a photomultiplier. The current required by the d.c. electromagnet to restore the mean plane of polarisation is automatically recorded. G. SKIRROW

3954. **Photo-electric gas analysis.** E. B. Offutt and L. V. Sorg [Standard Oil Co. (Indiana)]. U.S. Pat. 2,800,397 (23.7.1957).—The determination by a photo-electric cell of changes in optical density of a stain on a light-stable, gas-impermeable tape moving through a sampling and analysing instrument provides an automatic, continuous and permanent record of the concn. of reactive gases in an atmospheric sample. The tape is pretreated with a mixture of *M* Pb acetate and *M* Na acetate maintained at pH 6 and with a gelatin coating, dried, and reeled. The detection of H_2S in the range of 0 to 500 p.p.m. may be recorded directly and higher concn. may be recorded quant. through controlled dilution of the influent sample stream. The method and apparatus are used to detect H_2S in the atmosphere, especially at petroleum refineries. CHEM. ABSTR.

See also Abstract—**3760**, Spectrographic analysis of ores.

Thermal

3955. **A new design of melting-point apparatus.** H. J. Barber, D. P. Odell and W. R. Wragg (May & Baker Ltd., Dagenham, Essex). *Chem. & Ind.*, 1958, (6), 153–155.—The arrangement described and illustrated is designed to standardise rapid determinations of m.p. of organic compounds. The accuracy is ± 1° with a rate of heating of ⩾ 5° per min., and reproducibility is within 1° even at 335°. Three capillary glass tubes are housed in a metal block and are heated by a microflame (gas). Only one thermometer (borosilicate glass, 20° to 360°) is used, the lower part fitting closely into a hole in the block. The air space within the block is protected from draughts by a tightly fitting Pyrex-glass sleeve. Illumination and a Fulmar lens ensure a high standard of viewing. W. J. BAKER

3956. **New types of recording differential thermobalances.** P. L. Waters (Coal Res. Sect., C.S.I.R.O., North Ryde, N.S.W., Australia). *J. Sci. Instrum.*, 1958, **35** (2), 41–46.—Two thermobalances that automatically record both differential and cumulative weight losses are described. In one the sample is counter-balanced by the suspended electrode of a silver coulometer; the controlling current gives a measure of the rate of change of weight. In the second, a servo-motor rotates the head of a torsion balance, the rate of change of weight being proportional to the speed of the motor and to the voltage applied to it. This balance can record weight changes up to 0·5 g at a max. rate of 30 mg per min. G. SKIRROW

3957. **Differential thermal analysis of organic solids.** M. C. P. Varma (Gov. of Bihar, Patna, India). *J. Appl. Chem.*, 1958, **8** (2), 117–121.—With the apparatus previously described (Grimshaw *et al.*, *Trans. Brit. Ceram. Soc.*, 1945, **44**, 69), the endo- and exo-thermic effects associated with melting, boiling, sublimation, oxidation or decomposition of organic compounds were followed, and the m.p. and b.p. were determined to within ± 3°. Latent heats of fusion as calculated from endothermic peak areas are in agreement with known values. Results for catechol, D-camphor, *p*-nitrophenol, thiourea, sucrose, cryst. oxalic acid and other compounds are reported and discussed briefly. W. J. BAKER

Electrical

3958. **Quantitative microscopic ionophoresis and chromatography.** B. M. Turner (Turner's Lighter Spares & Components Ltd., Burroughs House, London, N.W.4). *Mikrochim. Acta*, 1958, (3), 305–318 (in English).—The procedure described can be applied to liquid films, 0·01 to 10 μ thick, on a polished glass slide or freshly-blown glass surface in a moist enclosure. The film contains the developing acid (HCl, formic acid, trichloroacetic acid) and additives, *e.g.*, Teepol, to control adsorption, ionic mobility or endosmosis. Drop-volume and film-thickness are measured by two-beam reflection interferometry, and the zones are detected by a modified "breath figure" test observed interferometrically. Zone areas are assumed to be quantitative. The cations are resolved by electrophoresis, chromatography, or both, with endosmosis providing the solvent flow; the film is then neutralised with NH_3 vapour and dried rapidly. As little as 1·6 to 4·0 × 10^{-3} pg of an inorganic cation can be quant. resolved in a run of 15 to 180 sec. (pg scale = 10^{-12} g scale). The lower limit of identification is ≃ 10^{-5} pg (≡ ≃ 63 × 10^3 atoms); electrophoresis can be 40 to 1000 times as sensitive as chromatography because of the greater zone-spread. W. J. BAKER

3959. **Reflectometric evaluation of electrophoresis bands. New design of apparatus for horizontal paper electrophoresis.** L. Strauch ("J. Stefan" Inst., Ljubljana, Yugoslavia). *Rep. "J. Stefan" Inst., Ljubljana*, 1957, **4**, 175–180 (in German).—Constructional details are described of an apparatus for horizontal paper electrophoresis in which the paper is stretched between two water-cooled surfaces. With this apparatus, protein fractions can be uniformly separated and the bands evaluated reflectometrically. G. BURGER

3960. **New method for the polarographic analysis of small amounts of flowing solutions.** V. Vojíř (Res. Inst. Org. Synth., Pardubice-Rybitví, Czechoslovakia). *Chem. Listy*, 1958, **52** (2), 335–336.—A hanging mercury drop on the lower part of the capillary tube is used as an indicator electrode, and a platinum wire wound round the lower part of the same capillary tube is a satisfactory reference electrode. A detailed description of the arrangement and manipulation is given. J. ŽÝKA

ABBREVIATIONS

Certain abbreviations in everyday use are not included in the following list. When any doubt might arise from the use of an abbreviation or symbol the word is printed in full.

alternating current	a.c.
ampere	amp.
Ångström unit	A
anhydrous	anhyd.
approximate, -ly	approx.
aqueous	aq.
atmospher-e, -ic	atm.
boiling-point	b.p.
British thermal unit	B.Th.U.
calorie (large)	kg-cal.
calorie (small)	g-cal.
centimetre	cm
coefficient	coeff.
concentrated	conc.
concentration	concn.
constant	const.
corrected	(corr.)
crystalline / crystallised	cryst.
cubic	cu.
current density	c.d.
cycles per second	c/s
density	ρ
density, relative	d or wt. per ml
dilute	dil.
direct current	d.c.
distilled	dist.
ethylenediaminetetra-acetic acid	EDTA
electromotive force	e.m.f.
equivalent	equiv.
gram	g
gram-molecule	mole
half-wave potential	$E_{\frac{1}{2}}$
hour	hr.
hydrogen ion exponent	pH
infra-red	i.r.
insoluble	insol.
international unit	i.u.
kilogram	kg
kilovolt	kV
kilowatt	kW
liquid	liq.
maxim-um, -a	max.
melting-point	m.p.
microgram	μg (not γ)
microlitre	μl
micromole	μmole
micron	μ
milliampere	mA
milli-equivalent	milli-equiv.
milligram	mg
millilitre	ml
millimetre	mm
millimicrogram	mμg
millimolar	mM
millivolt	mV
minute (time)	min.
molar (concentration)	M
molecul-e, -ar	mol.
normal (concentration)	N
optical rotation	α_D^t
ounce	oz
parts per million	p.p.m.
per cent.	%
per cent. (vol. in vol.)	% (v/v)
per cent. (wt. in vol.)	% (w/v)
per cent. (wt. in wt.)	% (w/w)
potential difference	p.d.
precipitate (as a noun)	ppt.
precipitated	pptd.
precipitating	pptg.
precipitation	pptn.
preparation	prep
qualitative, -ly	qual.
quantitative, -ly	quant.
recrystallised	recryst.
refractive index	n_D^t
relative band speed	R_{B}
relative humidity	r.h.
revolutions per minute	r.p.m.
saponification value	sap. val.
saturated calomel electrode	S.C.E.
second (time)	sec.
soluble	sol.
solution	soln.
specific gravity	sp. gr.
specific rotation	$[\alpha]_D^t$
square centimetre	sq. cm
standard temp. and pressure	s.t.p.
temperature	temp.
ultra-violet	u.v.
vapour density	v.d.
vapour pressure	v.p.
volt	V
volume	vol.
watt	W
wavelength	λ
weight	wt.

In addition, the following symbols may be used in conjunction with numerical values or in mathematical expressions—

greater than	$>$	less than	$<$
not greater than	$\ngtr$	not less than	$\nless$
is proportional to	$\propto$	of the order of, approximately	$\simeq$

The principal Pharmacopoeias are denoted by B.P., U.S.P. or D.A.B., together with the identifying roman numeral or year.

Valency states are represented by a superscript roman numeral, *e.g.*, Fe^{II}, Mo^{V}. Substances in the ionic state are represented by Na^+, Fe^{2+}, Fe^{3+}, etc., for cations and by Cl^-, SO_4^{2-}, PO_4^{3-}, etc., for anions.

additional mirror. By using a high-pressure mercury lamp the 265-mμ line can be isolated with an admixture of only 1·0% of stray light without the use of filters. E. KAWERAU

3953. **Automatic recording saccharimeter.** J. W. Gates (Nat. Phys. Lab., England). *Chem. & Ind.*, 1958, (7), 190–193.—A beam of plane-polarised light passes through a 10-cm DEDF glass rod that forms the core of an electromagnet. The 50 c.p.s. magnetic field induces an optical rotation alternating 3° either side of the unmodulated direction. The modulated beam passes through the sample cell and a second (5 cm) glass rod which forms the core of a d.c. electromagnet. After passing through an analyser, the beam is observed by a photomultiplier. The current required by the d.c. electromagnet to restore the mean plane of polarisation is automatically recorded. G. SKIRROW

3954. **Photo-electric gas analysis.** E. B. Offutt and L. V. Sorg (Standard Oil Co. (Indiana)). U.S. Pat. 2,800,397 (23.7.1957).—The determination by a photo-electric cell of changes in optical density of a stain on a light-stable, gas-impermeable tape moving through a sampling and analysing instrument provides an automatic, continuous and permanent record of the concn. of reactive gases in an atmospheric sample. The tape is pretreated with a mixture of *M* Pb acetate and *M* Na acetate maintained at pH 6 and with a gelatin coating, dried, and reeled. The detection of H_2S in the range of 0 to 500 p.p.m. may be recorded directly and higher concn. may be recorded quant. through controlled dilution of the influent sample stream. The method and apparatus are used to detect H_2S in the atmosphere, especially at petroleum refineries. CHEM. ABSTR.

See also Abstract—**3760**, Spectrographic analysis of ores.

Thermal

3955. **A new design of melting-point apparatus.** H. J. Barber, D. P. Odell and W. R. Wragg (May & Baker Ltd., Dagenham, Essex). *Chem. & Ind.*, 1958, (6), 153–155.—The arrangement described and illustrated is designed to standardise rapid determinations of m.p. of organic compounds. The accuracy is ± 1° with a rate of heating of 3–5° per min., and reproducibility is within 1° even at 335°. Three capillary glass tubes are housed in a metal block and are heated by a microflame (gas). Only one thermometer (borosilicate glass, 20° to 360°) is used, the lower part fitting closely into a hole in the block. The air space within the block is protected from draughts by a tightly fitting Pyrex-glass sleeve. Illumination and a Fulmar lens ensure a high standard of viewing. W. J. BAKER

3956. **New types of recording differential thermobalances.** P. L. Waters (Coal Res. Sect., C.S.I.R.O., North Ryde, N.S.W., Australia). *J. Sci. Instrum.*, 1958, **35** (2), 41–46.—Two thermobalances that automatically record both differential and cumulative weight losses are described. In one the sample is counter-balanced by the suspended electrode of a silver coulometer; the controlling current gives a measure of the rate of change of weight. In the second, a servo-motor rotates the head of a torsion balance, the rate of change of weight being proportional to the speed of the motor and to the voltage applied to it. This balance can record weight changes up to 0·5 g at a max. rate of 30 mg per min. G. SKIRROW

3957. **Differential thermal analysis of organic solids.** M. C. P. Varma (Gov. of Bihar, Patna, India). *J. Appl. Chem.*, 1958, **8** (2), 117–121.—With the apparatus previously described (Grimshaw *et al.*, *Trans. Brit. Ceram. Soc.*, 1945, **44**, 69), the endo- and exo-thermic effects associated with melting, boiling, sublimation, oxidation or decomposition of organic compounds were followed, and the m.p. and b.p. were determined to within ± 3°. Latent heats of fusion as calculated from endothermic peak areas are in agreement with known values. Results for catechol, D-camphor, *p*-nitrophenol, thiourea, sucrose, cryst. oxalic acid and other compounds are reported and discussed briefly. W. J. BAKER

Electrical

3958. **Quantitative microscopic ionophoresis and chromatography.** B. M. Turner (Turner's Lighter Spares & Components Ltd., Burroughs House, London, N.W.4). *Mikrochim. Acta*, 1958, (3), 305–318 (in English).—The procedure described can be applied to liquid films, 0·01 to 10 μ thick, on a polished glass slide or freshly-blown glass surface in a moist enclosure. The film contains the developing acid (HCl, formic acid, trichloroacetic acid) and additives, *e.g.*, Teepol, to control adsorption, ionic mobility or endosmosis. Drop-volume and film-thickness are measured by two-beam reflection interferometry, and the zones are detected by a modified "breath figure" test observed interferometrically. Zone areas are assumed to be quantitative. The cations are resolved by electrophoresis, chromatography, or both, with endosmosis providing the solvent flow; the film is then neutralised with NH_3 vapour and dried rapidly. As little as 1·6 to 4·0 × 10^{-3} pg of an inorganic cation can be quant. resolved in a run of 15 to 180 sec. (pg scale = 10^{-12} g scale). The lower limit of identification is $\simeq 10^{-3}$ pg (= $\simeq 63 \times 10^3$ atoms); electrophoresis can be 40 to 1000 times as sensitive as chromatography because of the greater zone-spread. W. J. BAKER

3959. **Reflectometric evaluation of electrophoresis bands. New design of apparatus for horizontal paper electrophoresis.** L. Strauch ("J. Stefan" Inst., Ljubljana, Yugoslavia). *Rep. "J. Stefan" Inst., Ljubljana*, 1957, **4**, 175–180 (in German).—Constructional details are described of an apparatus for horizontal paper electrophoresis in which the paper is stretched between two water-cooled surfaces. With this apparatus, protein fractions can be uniformly separated and the bands evaluated reflectometrically. G. BURGER

3960. **New method for the polarographic analysis of small amounts of flowing solutions.** V. Vojir (Res. Inst. Org. Synth., Pardubice-Rybitví, Czechoslovakia). *Chem. Listy*, 1958, **52** (2), 335–336.—A hanging mercury drop on the lower part of the capillary tube is used as an indicator electrode, and a platinum wire wound round the lower part of the same capillary tube is a satisfactory reference electrode. A detailed description of the arrangement and manipulation is given. J. ŽÝKA

ABBREVIATIONS

Certain abbreviations in everyday use are not included in the following list. When any doubt might arise from the use of an abbreviation or symbol the word is printed in full.

alternating current	a.c.
ampere	amp.
Ångström unit	A
anhydrous	anhyd.
approximate, -ly	approx.
aqueous	aq.
atmospher-e, -ic.	atm.
boiling-point	b.p.
British thermal unit	B.Th.U.
calorie (large)	kg-cal.
calorie (small)	g-cal.
centimetre	cm
coefficient	coeff.
concentrated	conc.
concentration	concn.
constant	const.
corrected	(corr.)
crystalline / crystallised	cryst.
cubic	cu.
current density	c.d.
cycles per second	c/s
density	ρ
density, relative	d or wt. per ml
dilute	dil.
direct current	d.c.
distilled	dist.
ethylenediaminetetra-acetic acid	EDTA
electromotive force	e.m.f.
equivalent	equiv.
gram	g
gram-molecule	mole
half-wave potential	$E_{\frac{1}{2}}$
hour	hr.
hydrogen ion exponent	pH
infra-red	i.r.
insoluble	insol.
international unit	i.u.
kilogram	kg
kilovolt	kV
kilowatt	kW
liquid	liq.
maxim-um, -a	max.
melting-point	m.p.
microgram	μg (not γ)
microlitre	μl
micromole	μmole
micron	μ
milliampere	mA
milli-equivalent	milli-equiv.
milligram	mg
millilitre	ml
millimetre	mm
millimicrogram	mμg
millimolar	mM
millivolt	mV
minute (time)	min.
molar (concentration)	M
molecul-e, -ar	mol.
normal (concentration)	N
optical rotation	α_{λ}^{t}
ounce	oz
parts per million	p.p.m.
per cent.	%
per cent. (vol. in vol.)	% (v/v)
per cent. (wt. in vol.)	% (w/v)
per cent. (wt. in wt.)	% (w/w)
potential difference	p.d.
precipitate (as a noun)	ppt.
precipitated	pptd.
precipitating	pptg
precipitation	pptn.
preparation	prep
qualitative, -ly	qual.
quantitative, -ly	quant.
recrystallised	recryst.
refractive index	n_{λ}^{t}
relative band speed	R_{B}
relative humidity	r.h.
revolutions per minute	r.p.m.
saponification value	sap. val.
saturated calomel electrode	S.C.E.
second (time)	sec.
soluble	sol.
solution	soln.
specific gravity	sp. gr.
specific rotation	$[\alpha]_{\lambda}^{t}$
square centimetre	sq. cm
standard temp. and pressure	s.t.p.
temperature	temp.
ultra-violet	u.v.
vapour density	v.d.
vapour pressure	v.p.
volt	V
volume	vol.
watt	W
wavelength	λ
weight	wt.

In addition, the following symbols may be used in conjunction with numerical values or in mathematical expressions—

greater than	$>$	less than	$<$
not greater than	$\ngtr$	not less than	$\nless$
is proportional to	$\propto$	of the order of, approximately	$\simeq$

The principal Pharmacopoeias are denoted by B.P., U.S.P. or D.A.B., together with the identifying roman numeral or year.

Valency states are represented by a superscript roman numeral, *e.g.*, Fe^{II}, Mo^{V}. Substances in the ionic state are represented by Na^{+}, Fe^{3+}, Fe^{2+}, etc., for cations and by Cl^{-}, SO_4^{2-}, PO_4^{3-}, etc., for anions.

Volume 5. No. 11, Abstracts 3584-3960 November, 1958

ANALYTICAL ABSTRACTS

A PUBLICATION OF
THE SOCIETY FOR ANALYTICAL CHEMISTRY

EDITORIAL COMMITTEE

Chairman: R. C. Chirnside

Members: B. S. Cooper, B. A. Ellis, C. H. R. Gentry, C. A. Johnson, I. MacIntyre, B. J. Walby, W. A. Waygood and the President and Honorary Officers

President of the Society: J. H. Hamence

Hon. Secretary of the Society: R. E. Stuckey

Hon. Assistant Secretary of the Society: S. A. Price

Hon. Treasurer of the Society: A. J. Amos

Editor: Norman Evers, B.Sc., Ph.D., F.R.I.C.

Assistant Editor: Mrs. H. I. Fisk, B.Sc.

CONTENTS

	Abstract
General Analytical Chemistry	3584
Inorganic Analysis	3600
Organic Analysis	3762
Biochemistry	
Blood, Bile, Urine, etc.	3818
Drugs	3873
Food	3893
Sanitation	3917
Agriculture and Plant Biochemistry	3923
General Technique and Laboratory Apparatus	
General	3938
Optical	3951
Thermal	3955
Electrical	3958

Printed and Published for the Society for Analytical Chemistry by W. Heffer & Sons Ltd., Cambridge, England.
Communications to be addressed to the Editor, Norman Evers, 14, Belgrave Square, London, S.W.1.
Enquiries about advertisements should be addressed to Walter Judd Ltd., 47, Gresham Street, London, E.C.2.
Entered as Second Class at New York, U.S.A., Post Office